

**NASA CONTRACTOR  
REPORT**



NASA CR  
2.1

0061029



NASA CR-1849

LOAN COPY: RETURN TO  
AFV L (DOGL)  
KIRTLAND AFB, N. M.

**SENSITIVITY OF THE CREEP-RUPTURE  
PROPERTIES OF WASPALOY SHEET  
TO SHARP-EDGED NOTCHES IN THE  
TEMPERATURE RANGE OF 1000° TO 1400° F**

*by David J. Wilson and James W. Freeman*

*Prepared by*

UNIVERSITY OF MICHIGAN

Ann Arbor, Mich.

*for*



0061029

1. Report No. <b>NASA CR 1849</b>		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle <b>SENSITIVITY OF THE CREEP-RUPTURE PROPERTIES OF WASPALOY SHEET TO SHARP-EDGED NOTCHES IN THE TEMPERATURE RANGE OF 1000° TO 1400° F</b>				5. Report Date <b>June 1971</b>	
				6. Performing Organization Code	
7. Author(s) <b>David J. Wilson and James W. Freeman</b>				8. Performing Organization Report No. <b>04368-17-T</b>	
9. Performing Organization Name and Address <b>University of Michigan Ann Arbor, Michigan</b>				10. Work Unit No.	
				11. Contract or Grant No. <b>NGL-23-005-005</b>	
12. Sponsoring Agency Name and Address <b>National Aeronautics and Space Administration Washington, D.C. 20546</b>				13. Type of Report and Period Covered <b>Contractor Report</b>	
				14. Sponsoring Agency Code	
15. Supplementary Notes					
16. Abstract <p>Time dependent notch sensitivity was observed in Waspalloy sheet between 900° and 1300° F. It occurred in notched specimens loaded below their yield strength when tests of smooth specimens showed that small amounts of creep consumed large fractions of the creep-rupture life. "Overaging" eliminated the time-dependent notch sensitivity and produced a change in the dislocation mechanism from shearing to by-passing the gamma prime particles.</p>					
17. Key Words (Suggested by Author(s)) <b>Heat resistant alloys; Nickel alloys; Waspalloy; Creep properties; Notch sensitivity; Dislocations</b>				18. Distribution Statement <b>Unclassified - unlimited</b>	
19. Security Classif. (of this report) <b>Unclassified</b>		20. Security Classif. (of this page) <b>Unclassified</b>		21. No. of Pages <b>175</b>	
				22. Price* <b>\$3.00</b>	



## **FOREWORD**

The research described in this report was conducted by the University of Michigan under NASA grant NGL-23-005-005. Mr. John C. Freche of the Lewis Research Center Materials and Structures Division was the NASA Project Manager. The report was originally issued as University of Michigan report 04368-17-T.



# TABLE OF CONTENTS

Page

INTRODUCTION . . . . .	1
BACKGROUND . . . . .	3
EXPERIMENTAL MATERIAL . . . . .	5
EXPERIMENTAL DESIGN . . . . .	6
Heat Treatment Variations . . . . .	6
Notched Specimen Geometry . . . . .	9
Significance of "Yielding" . . . . .	10
EXPERIMENTAL PROCEDURES . . . . .	11
Reduction of 0.310-inch Plate and 0.050-inch Thick Sheet . . . . .	11
Heat Treatment . . . . .	11
Test Specimens . . . . .	12
Smooth Specimens . . . . .	12
Notched Specimens . . . . .	12
Testing Methods . . . . .	13
Hardness . . . . .	13
Tensile Tests . . . . .	13
Creep-Rupture Tests . . . . .	14
Structural Examination . . . . .	15
Optical Microscopy . . . . .	15
Electron Microscopy . . . . .	16
X-Ray Diffraction . . . . .	18
INFLUENCE OF HEAT TREATMENT ON THE MECHANICAL CHARACTERISTICS . . . . .	19
Exploratory Tests At 1000° And 1200°F . . . . .	19
Selected Heat Treated Conditions . . . . .	23
Tensile and Creep-Rupture Strength Characteristics . . . . .	24

	Page
Ductility in Creep-Rupture Tests. . . . .	26
FRACTURE CHARACTERISTICS. . . . .	28
STRESS RELAXATION. . . . .	35
Relaxation By "Yielding" . . . . .	35
Relaxation By Creep . . . . .	37
Materials Exhibiting Time-Dependent Notch Sensitivity .	38
Materials Which Did Not Exhibit Time-Dependent Notch Sensitivity . . . . .	40
Consequences Of The Correlation Between Deformation Characteristics And The Time-Dependent Notch Sensitivity . . . . .	43
MICROSTRUCTURAL FEATURES CONTRIBUTING TO TIME-DEPENDENT NOTCH SENSITIVITY . . . . .	45
Interactions Of Dislocations With Gamma Prime . . . . .	47
Materials Solution Treated At 1900°, 1975° and 2150°F And Aged 16 Hours At 1400°F . . . . .	47
Materials Solution Treated at 1900°, 1975° and 2150°F And Aged 10 Hours At 1700°F . . . . .	50
Materials Heat Treated 1/2 Hour At 1975°F Plus 24 Hours At 1550°F, 1/2 Hour At 1975°F Plus 24 Hours At 1550°F Plus 16 Hours At 1400°F, And 4 Hours At 1975°F Plus 24 Hours At 1550°F Plus 16 Hours At 1400°F . . . . .	52
Materials Solution Treated At 1825°F . . . . .	53
Grain Boundary Structures. . . . .	55
Grain Boundary Precipitates . . . . .	55
Carbide Phase Identification By X-Ray Diffraction . . . . .	56
Orientation Relationships Of Grain Boundary Carbide . . . . .	56
Gamma Prime Depletion . . . . .	57
Correlation Of The Time-Dependent Notch Sensitivity With The Dislocation Structure . . . . .	58
Yield Strengths. . . . .	59
Deformation At Short Times . . . . .	60
Accumulation of Damage . . . . .	61
SUMMARY OF RESULTS . . . . .	65

	Page
APPENDIX I. TIME-TEMPERATURE RUPTURE CHARACTERISTICS. . . . .	67
APPENDIX II. INFLUENCE OF SHEET THICKNESS ON THE NOTCH SENSITIVITY . . . . .	73
REFERENCES . . . . .	76



# LIST OF TABLES

Table	Page
1 Tensile Properties of 0.026-inch Thick Sheet at 1000°F .	78
2 Smooth Specimen Tensile and Creep Rupture Properties at 1000° and 1200°F for 0.026-Inch Thick Sheet. . .	79
3 Notched Specimen Tensile and Rupture Properties at 1000° and 1200°F for 0.026-inch Thick Sheet . . .	80
4 Tensile Properties of 0.026-inch Thick Sheet at 1000°, 1200° and 1400°F . . . . .	81
5 Smooth and Notched Specimen Tensile and Creep Rupture Properties at 900°F to 1400°F for 0.026-Inch Thick Sheet Heat Treated 1/2 Hour at 1825°F Plus 16 Hours at 1400°F . . . . .	82
Smooth and Notched Specimen Tensile and Creep-Rupture Properties at 1000° to 1400°F for 0.026-inch Thick Sheet . .	83-87
6. Solution treated 1/2 hour at 1975°F and aged 16 hours at 1400°F . . . . .	83
7. Solution treated 1/2 hour at 1975°F and aged 10 hours at 1700°F . . . . .	84
8. Solution treated 1/2 hour at 1975°F and aged 24 hours at 1550°F . . . . .	85
9. Solution treated 1/2 hour at 1975°F and aged 24 hours at 1550°F plus 16 hours at 1400°F . . .	86
10. Solution treated 4 hours at 1975°F and aged 24 hours at 1550°F plus 16 hours at 1400°F . . .	87
11 Smooth and Notched Specimen Creep-Rupture Properties at 1000° to 1400°F for 0.026-inch Thick Sheet Annealed and Aged 16 Hours at 1400°F . . . . .	88
12 Smooth and Notched Specimen Creep-Rupture Properties at 1000° to 1400°F for 0.026-inch Thick Sheet Annealed and Aged 16 Hours at 1400°F. Smooth Specimens Pre- Strained Small Amounts and Notched Specimens Loaded to 115 ksi Prior to Creep-Rupture Testing . . . .	89

Table	Page
13 X-Ray Diffraction Data of Extracted Residues for Various Heat Treated Conditions . . . . .	90
14 Grain Sizes of Waspaloy Sheets in the As-Heat Treated Conditions . . . . .	91
15 Summary of Optimized Stress-Rupture Parameter Constants for 0.026-inch Thick Sheet in Heat Treated Conditions . . . . .	92
16 Tensile Properties at 1000°, 1200° and 1400°F of Sheets 0.050 and 0.026-inches Thick Heat Treated 1/2 Hour at 1975°F and Aged 16 Hours at 1400°F . . . . .	93
17 Smooth and Notched Specimen Tensile and Creep Rupture Properties at 1000° to 1400°F for 0.050-inch Thick Sheet Heat Treated 1/2 Hour at 1975°F Plus 16 Hours at 1400°F . . . . .	94
18 Notched Specimen Rupture Properties at 1000° to 1200° F for 0.026-inch Thick Sheet Mechanically Thinned from 0.050-inch Thick Material and Subsequently Heat Treated 1/2 Hour at 1975°F Plus 16 Hours at 1400°F . . . . .	95
19 Smooth and Notched Specimen Creep Rupture Properties at 1000° to 1400°F for 0.013 to 0.075-inch Thick Sheets Heat Treated 1/2 Hour at 1975°F Plus 16 Hours at 1400°F . . . . .	96

## LIST OF FIGURES

Figure	Page
1    Stress versus Rupture Time Data at 1000° and 1200°F Obtained from Smooth and Edge-Notched Specimens of 0.026-inch Thick Waspaloy Sheet in the Annealed and Aged Condition . . . . .	97
2    Types of Test Specimens . . . . .	98
Stress Versus Rupture Time Data at 1000° and 1200°F Obtained from Smooth and Edge-Notched Specimens of 0.026-inch Thick Sheet . . . . .	
3.    Solution Treated 1/2 Hour at 1825°F and Aged . . .	99
4.    Solution Treated 1/2 Hour at 1900°F and Aged . . .	100
5.    Solution Treated 1/2 Hour at 1975°F and Aged . . .	101
6.    Solution Treated 1/2 Hour at 2150°F and Aged . . .	102
7    Stress Versus Rupture Time Data at 1000° and 1200°F Obtained from Smooth Specimens of 0.026-inch Thick Sheet in the Heat Treated Condition . . . . .	103
8    Stress Versus Rupture Time Data at 1000° and 1200°F Obtained from Notched Specimens of 0.026-inch Thick Sheet in the Heat Treated Condition . . . . .	104
9    Stress Versus Minimum Creep Rate Behavior at 1000° and 1200°F for 0.026-inch Thick Sheet in the Heat Treated Condition . . . . .	105
10   Elongation Versus Rupture Time Data at 1000° and 1200°F for Smooth Specimens of 0.026-inch Thick Sheet in the Heat Treated Condition . . . . .	106
11   Reduction in Area Versus Rupture Time Data at 1000° and 1200°F for Smooth Specimens of 0.026-inch Thick Sheet in the Heat Treated Condition . . . . .	107
12   Stress Versus Rupture Time Data at Temperatures from 900° to 1400°F Obtained from Smooth and Notched Specimens of 0.026-inch Thick Sheet Solution Treated 1/2 Hour at 1825°F and Aged 16 Hours at 1400°F . . .	108

Figure	Page
Stress Versus Rupture Time Data at Temperatures from 1000° to 1400°F Obtained from Smooth and Notched Specimens of 0.026-inch Thick Sheet . . . . .	109 - 111
13. Solution Treated 1/2 Hour at 1975°F and Aged at 1400°F . . . . .	109
14. Solution Treated 1/2 Hour at 1975°F and Aged at 1700°F . . . . .	110
15. Solution Treated at 1975°F and Aged . . . . .	111
16 Stress Versus Rupture Time Data at Temperatures from 1000° to 1400°F Obtained from Smooth and Notched Specimens of 0.026-inch Thick Sheet in the Heat Treated Condition . . . . .	112 - 114
17 Elongation Versus Rupture Time Data at Temperatures from 1000° to 1400°F for Smooth Specimens of 0.026-inch Thick Sheet in the Heat Treated Condition	115 - 117
18 Fracture Surfaces of Ruptured Specimens . . . . .	118
19 Optical Photomicrographs Showing the Fracture Path Across a Specimen . . . . .	119, 120
20 Photomicrograph Showing an Intergranular Crack Developing from a Machined Notch . . . . .	121
21 Photomicrograph Showing Subsidiary Cracking in the Region of the Major Intergranular Crack . . . . .	121
Intergranular Crack Length Versus Initial Loading Stress for Waspaloy . . . . .	122, 123
22. Solution Treated 1/2 Hour at 1975°F and Aged at 1400°F . . . . .	122
23. Solution Treated 1/2 Hour at 1975°F and Aged at 1700°F . . . . .	123
24 Intergranular Crack Length Versus Loading Stress for Sheet Materials in the Heat Treated Condition. Data from the Fractures of Smooth Specimens Tensile and Rupture Tested at Temperatures from 1000° to 1400°F	124

Figure	Page
25 Intergranular Crack Length <sup>1</sup> Versus Loading Stress for Sheet Materials in the Heat Treated Conditions. Data from the Fractures of Notched Specimens Tensile and Rupture Tested at Temperatures from 1000° to 1400°F .	125
26 Intergranular Crack Length Versus Time for a Notched Specimen Heat Treated 1/2 Hour at 1975°F Plus 16 Hours at 1400°F and Tested at 1000°F at 85 ksi . . .	126
27, 28 Photomicrographs Showing Subsidiary Cracking In Smooth Specimens . . . . .	127
29 Optical Photomicrographs of a Notched Specimen Polished to Remove the Surface Oxidized Layer. (Heat Treated 1/2 Hour at 1975°F Plus 16 Hours at 1400°F, Exposed for 90 Percent of its Rupture Life at 1000°F at 80 ksi) . . . . .	128
30 The Effect of Prestraining on the Time-Temperature Dependence of the Rupture Strengths of Smooth and Notched Specimens of 0.026-inch Thick Sheet Annealed and Aged 16 Hours at 1400°F. . . . .	129
31 Iso-Creep Strain Curves of Life Fraction Versus Stress at Temperatures from 950° to 1400°F for 0.026-inch Thick Sheet Solution Treated 1/2 Hour at 1825°F and Aged 16 Hours at 1400°F . . . . .	130
Iso-Creep Strain Curves of Life Fraction Versus Stress at Temperatures from 1000° to 1400°F for 0.026-inch Thick Sheet . . . . .	131 - 133
32. Solution Treated 1/2 Hour at 1975°F and Aged at 1400°F. . . . .	131
33. Solution Treated 1/2 Hour at 1975°F and Aged at 1700°F. . . . .	132
34. Solution Treated 1/2 Hour at 1975°F and Aged at 1550°F and at 1400°F . . . . .	133
35 Optical Photomicrographs for Various Heat Treated Conditions . . . . .	134, 135

Figure	Page
36 Replica Electron Micrographs of Waspaloy Sheet in the As-Heat-Treated Condition . . . . .	136, 137
Transmission Electron Micrographs of Waspaloy Heat Treated 1/2 Hour at 1975°F and Aged 16 Hours at 1400°F . . . . .	
37. Tensile Tested at 1000°F . . . . .	138
38. Creep-Rupture Tested at 135 ksi at 1000°F . . . . .	139
39. Strained 2.3 Percent at 1000°F . . . . .	140
40. Creep-Rupture Tested at 1200° and 1300°F . . . . .	141
41. Tensile Tested at 1400°F . . . . .	142
42. Creep-Rupture Tested at 38 ksi at 1400°F . . . . .	143
Transmission Electron Micrographs of Waspaloy Heat Treated 1/2 Hour at 1975°F and Aged 10 Hours at 1700°F . . . . .	
43. Tensile Tested at 1000°F . . . . .	144
44. Strained Approximately 1, 2.5 and 6% at 1000°F . . . . .	145
45. Creep-Rupture Tested at 1000° and 1200°F . . . . .	146
46. Tensile Tested at 1400°F . . . . .	147
47. Creep-Rupture Tested at 38 ksi at 1400°F . . . . .	148
48 Effect of Aging Exposures at 1325°, 1400°, 1550° and 1700°F on the Diamond Pyramid Hardness of Several Selected Heat Treatments of Waspaloy Sheet . . . . .	149
49 Thin-Foil Electron Micrographs of Waspaloy Heat Treated 1/2 Hour at 1975°F and Aged 24 Hours at 1550°F and Strained About 2% at 1000°F . . . . .	150
50 Transmission Electron Micrographs of Waspaloy Heat Treated 4 Hours at 1975°F and Aged 24 Hours at 1550° Plus 16 Hours at 1400°F and Strained Approximately 2% at 1000°F . . . . .	150

Figure	Page
51 Thin-Foil Electron Micrographs of Waspaloy Heat Treated 1/2 Hour at 1975°F and Aged 24 Hours at 1550°F and Creep Rupture Tested at 80 ksi at 1200°F .	151
52 Thin-Foil Electron Micrographs of Waspaloy Heat Treated 4 Hours at 1975°F and Aged 24 Hours at 1550°F Plus 16 Hours at 1400°F and Creep-Rupture Tested at 80 ksi at 1200°F. . . . .	152
53 Transmission Electron Micrographs of Waspaloy Heat Treated 1/2 Hour at 1975°F and Aged 24 Hours at 1550°F and Creep Rupture Tested at 38 ksi at 1400°F .	153
54 Transmission Electron Micrographs Showing $\gamma'$ Size Distributions of Waspaloy Heat Treated 1/2 Hour at 1825°F and Aged 16 Hours at 1400°F and Creep Rupture Tested at 1100° and 1300°F . . . . .	154
Transmission Electron Micrographs of Waspaloy Heat Treated 1/2 Hour at 1825°F and Aged 16 Hours at 1400°F .	155, 156
55. Creep-Rupture Tested at 1000° and 1100°F . . . . .	155
56. Creep-Rupture Tested at 1200° and 1300°F . . . . .	156
57, 58 Electron Micrographs Showing $\gamma'$ and Carbide Precipitates in the Grain Boundary . . . . .	157
59 Selected Area Diffraction Pattern of a Grain Boundary Showing Identical Orientations of $M_{23}C_6$ and the Matrix on One Side of the Boundary . . . . .	158
60 Transmission Electron Micrographs of Waspaloy Showing Zones Denuded of $\gamma'$ that Formed During Creep-Rupture Exposure . . . . .	159
61 Time-Temperature Dependence of the Rupture Strengths at Temperatures from 900° to 1400°F of Smooth and Notched Specimens of 0.026-inch Thick Sheet Heat Treated 1/2 Hour at 1825°F and Aged 16 Hours at 1400°F.	160

Figure	Page
Time-Temperature Dependence of the Rupture Strengths at Temperatures from 1000° to 1400°F of Smooth and Notched Specimens of 0.026-inch Thick Sheet . . . . .	161 - 163
62. Solution Treated 1/2 Hour at 1975°F and Aged at 1400°F . . . . .	161
63. Solution Treated 1/2 Hour at 1975°F and Aged at 1550°F . . . . .	162
64. Solution Treated 1/2 Hour at 1975°F and Aged at 1700°F . . . . .	163
65 Time-Temperature Dependence of the Rupture Strengths at Temperatures from 1000° to 1400°F of Smooth Specimens of 0.026-inch Thick Sheet in the Heat Treated Conditions . . . . .	164
66 Time-Temperature Dependence of the Rupture Strengths at Temperatures of 1000° and 1200°F of Smooth Specimens of 0.026-inch Thick Sheet in the Heat Treated Conditions . . . . .	165
67 Time-Temperature Dependence of the Rupture Strengths at Temperatures from 900° to 1400°F of Notched Specimens of 0.026-inch Thick Sheet Heat Treated 1/2 Hour at 1825°F and Aged, and 1/2 Hour at 1975°F and Aged . . . . .	166
68 Iso-Stress Curves of Rupture Time Versus Temperature for Notched Specimens of 0.026-inch Thick Sheet Heat Treated 1/2 Hour at 1825°F and Aged 16 Hours at 1400°F .	167
69 Iso-Stress Curves of Rupture Time Versus Reciprocal Absolute Temperature for Notched Specimens of 0.026-Inch Thick Sheet Heat Treated 1/2 Hour at 1825°F and Aged 16 Hours at 1400°F . . . . .	168
70 Stress Versus Rupture Time Data at Temperatures From 1000° to 1400°F for Notched and Smooth Specimens of 0.050 and 0.026-inch Thick Sheets Heat Treated 1/2 Hour at 1975°F and Aged 16 Hours at 1400°F . . . . .	169



- 71 Iso-Creep Strain Curves of Life Fraction Versus Stress  
at Temperatures from 1000° to 1400°F for 0.050-inch  
Thick Sheet Solution Treated 1/2 Hour at 1975°F and  
Aged 16 Hours at 1400°F . . . . . 170
- 72 Stress Versus Rupture Time Data at Temperatures from  
1000° to 1400°F for Notched Specimens of 0.050 and  
Mechanically Thinned 0.026-inch Thick Sheets Heat  
Treated 1/2 Hour at 1975°F and Aged 16 Hours at 1400°F . 171

## SUMMARY

A study was made of the time-dependent edge-notch sensitivity known to occur at 1000° and 1200°F for the superalloy sheet materials, Waspaloy, Rene' 41 and Inconel 718. Since the occurrence of this phenomenon could limit the utilization of nickel-base heat resistant alloys for service at intermediate test temperatures, it was important to establish the scope and cause of the problem. This in turn was expected to enable prediction of whether similar behavior could be expected for other alloys.

The experimental program utilizing Waspaloy was based on the concept that the time-dependent notch sensitivity would be dependent on particular mechanical characteristics and/or microstructural features. Heat treatments were selected to provide a wide range of these variables. Tensile and creep-rupture characteristics, including the notch sensitivity were evaluated for the most part at temperatures from 1000° to 1400°F using 0.026-inch thick sheet. Limited tests were also conducted for sheets varying in thickness from 0.013 to 0.075-inch. The microstructural features were studied for the as-heat treated materials and for tested specimens. Particular emphasis was directed at the determination of the nature of the interaction of dislocations with the gamma prime precipitate particles.

Necessary conditions for time-dependent notch sensitivity were (i) the notched specimen loads had to be below the approximate 0.2 percent smooth specimen offset yield strength; and (ii) test data from

smooth specimens had to indicate that small amounts of creep used up large fractions of creep-rupture life. No reasons were evident why these criteria will not prove applicable to any alloy.

Sheet thickness variation from 0.013 to 0.075-inch did not influence the results. Time-dependent notch sensitivity was observed at test temperatures from 900° to 1300°F. Its occurrence in this range was dependent on the heat treatment. "Overaging" eliminated the time-dependent notch sensitivity. Optimum combination of smooth and notched specimen strengths for material solution treated at 1975°F were obtained by aging 24 hours at 1550°F.

The dislocation motion mechanism varied with the heat treatment and test conditions. Dislocations sheared gamma prime particles smaller than a critical size. Particles larger than the critical were by-passed by dislocations. The former mechanism promoted the deformation-time characteristics that gave rise to the time-dependent notch sensitivity. In consequence, an apparent correlation existed between the notch sensitive behavior and the dislocation mechanism operative. Further research is necessary to determine the generality or limitations of this correlation.

## INTRODUCTION

This investigation is a study of the scope and causes of severe time-dependent edge-notch sensitivity that can occur for nickel-base superalloy sheet materials at 1000° and 1200°F. These temperatures are low enough so that the design strengths would normally be based on the time-independent smooth specimen tensile and yield strengths rather than time-dependent creep-rupture properties. For the alloys for which this behavior was discovered (ref. 1, 2) time-dependent failures of edge-notched specimens at short times due to exposure at relatively low stresses and low temperatures were entirely unexpected since the smooth specimen rupture life under these conditions was practically infinite and the ratios of notched to smooth specimen strengths (N/S ratio) in tensile tests were reasonably high. In addition, there was no reason to expect that the alloys would undergo damaging microstructural changes at these temperatures and especially not in the time periods involved.

Once it was established that these materials suffered a severe time-dependent notch sensitivity it became important to establish the scope and cause of the problem for the following reasons:

- (1) The utilization of nickel-base heat resistance alloys for service application at intermediate temperatures (i. e. , between temperatures where short time characteristics and creep behavior control design) could be severely limited by the time-dependent notch sensitivity. Therefore, developing the ability to predict the

notch sensitive behavior and also, if possible, to reduce or eliminate it by heat treatment, became important.

- (2) Once the cause of the time-dependent notch sensitivity is established, it should be possible to predict whether similar behavior should be expected for other alloys. Conceivably, brittle behavior observed at intermediate temperatures and attributed to other causes, may in fact prove to be due to the same type of notch sensitivity.

## BACKGROUND

Extensive research has been carried out by many laboratories to evaluate the potential usefulness of various superalloys in sheet form for construction of the Supersonic Transport (SST). Rene' 41, Waspaloy and Inconel 718 were determined to have the greatest potential for skin use in the Mach 3 transport (ref. 3). The selection was based on smooth and notched specimen tensile tests from -110°F to 650°F carried out after 1000 hours at 650°F and 40,000 psi (a reasonable estimate of the design stress for the SST). The results were evaluated on the basis of stability of these properties, especially the retention of resistance to catastrophic crack propagation, subsequent to exposure.

The three superalloys selected were subsequently subjected to study in greater detail with the objective of determining their upper temperature of usefulness under the design conditions expected for the SST (ref. 2, 3). This involved carrying out exposures under a stress of 40,000 psi for 1000 hours at 800°, 1000° and 1200°F, with the intention of determining whether the exposures were detrimental to the short time tensile properties. However, several of the notched specimens failed unexpectedly in less than 1000 hours during exposure at 1000°F. When the specimens which survived exposure were subsequently tensile tested, no significant change in properties were found.

The failures of notched specimens during exposure at 1000°F

suggested that the notches caused a marked time-dependent effect on strength even though the temperature was far lower than where such behavior was expected. Subsequent characterization of the sensitivity to edge-notches did, in fact, reveal a marked time-dependence at 1000° and 1200°F. Time-dependent notch sensitivity was defined as occurring when the notched to smooth rupture strength ratios were smaller than the ratio established by tensile tests. A typical example is presented as Figure 1 (notched to smooth tensile ratio of about 0.8). This behavior was shown to occur for sharp notches, designed to simulate cracks; however, the research also demonstrated that in many cases the notches did not have to be very sharp to induce the premature fractures.

The accumulated evidence raised the likelihood that the unusual sensitivity of these alloys in sheet form to stress concentration could limit their application. Consequently, a study of the scope and cause of the notch sensitivity was carried out. The research utilized Waspaloy, a gamma prime precipitation hardened nickel-base super-alloy. However, it was expected that the results would act as a guide in determining the probability of similar behavior in other alloys, as well as ensuring proper recognition of the potential ramifications of the existence of the phenomenon.

## EXPERIMENTAL MATERIAL

The Waspaloy used in this study was received\* as sheet and plate in the following conditions:

<u>U. M. Code</u>	<u>Thickness (inches)</u>	<u>Condition</u>
W-I	0.026	Solution treated and cold reduced 23 to 25 percent
W-II	0.050	Solution treated and cold reduced 23 to 25 percent
W-III	0.310	Hot rolled; stress relieved at 2000°F and air cooled
W-IV	0.026	Annealed

The solution treatment being used for sheet at the time they were received was an air cool from 1975°F. This is most probably the "anneal" used for the W-IV material. The 2000°F treatment for the W-III material was not only a stress relief, but also a solution treatment.

These materials were reported by the suppliers to have the following chemical compositions (weight percent):-

<u>Element</u>	<u>W-I &amp; W-III</u>	<u>W-II</u>	<u>W-IV</u>
Nickel (by diff.)	57.95	57.90	57.90
Chromium	19.33	19.47	19.63
Cobalt	13.52	13.72	13.49
Molybdenum	4.16	4.25	4.26
Titanium	2.95	2.89	2.99
Aluminum	1.35	1.39	1.40
Carbon	0.06	0.08	0.06
Boron	0.005	0.006	0.005
Zirconium	0.03	0.02	0.03
Iron	0.55	0.18	0.30
Sulphur	0.007	0.007	0.007
Manganese	<0.01	0.01	0.04
Silicon	0.05	0.07	0.07
Copper	0.03	<0.01	-

\* The materials were supplied by the General Electric and Union Carbide Corporations.



## EXPERIMENTAL DESIGN

The dual objectives of the program, to determine the scope and the cause of the time-dependent notch sensitivity, are intimately related and, therefore, were incorporated jointly into the experimental design. To simplify presentation, the experimental design has been subdivided into the following sections:

### Heat Treatment Variations

Stage 1 - The experimental program was based on the concept that the time-dependent notch sensitivity would be closely related to certain mechanical characteristics and/or microstructural features. Thus, in seeking to determine the scope and causes of the notch sensitive behavior, the research program utilized heat treatment variations which were selected to permit variations in significant microstructural features. The heat treatments chosen were also expected to result in a wide range of mechanical characteristics.

Four solution treatments of 0.026-inch thick cold worked material were selected for exploratory testing in the initial stage of the investigation. Half-hour solution treatments at 2150°, 1975°, 1900° and 1825°F were expected to provide variations in grain size and the degree of solution of the carbide and gamma prime phases. It should be noted that although these higher temperature exposures are referred to throughout this paper as "solution treatments", the use of this designation does not necessarily reflect complete solution

of all constituent phases.

Two separate aging treatments, 16 hours at 1400°F and 10 hours at 1700°F were applied after each of the solution treatments. These were designed to produce  $\gamma'$  particles whose average size would vary by an order of magnitude. The aging treatment at 1400°F was chosen so that, when applied with the 1975°F heat treatment, the combination would be typical of industrial practice.

Tensile tests at 1000°F and creep-rupture tests at 1000° and 1200°F of edge-notched and smooth specimens were used to characterize the mechanical properties. The microstructural features of the as-heat treated materials and tested specimens were evaluated using optical and electron metallography. X-ray studies were also carried out for the as-heat treated materials.

Stage 2 - Hardness tests were utilized to check and extend the microstructural results regarding the response of heat treated materials to solution and aging treatment variations. Materials heat treated (a) 1/2 hour at 1825°F (b) 1/2 hour at 1975°F (c) 4 hours at 1975°F and (d) 4 hours at 1975°F plus 24 hours at 1550°F were hardness tested after aging (from 1 to 240 hours) at temperatures from 1325° to 1700°F.

Stage 3 - The following heat treatments were selected for detailed evaluation:

- (a) 1/2 hour at 1825°F plus 16 hours at 1400°F.
- (b) 1/2 hour at 1975°F plus 16 hours at 1400°F.
- (c) 1/2 hour at 1975°F plus 10 hours at 1700°F.

(d) 1/2 hour at 1975°F plus 24 hours at 1550°F.

(e) 1/2 hour at 1975°F plus 24 hours at 1550°F plus 16 hours at 1400°F.

(f) 4 hours at 1975°F plus 24 hours at 1550°F plus 16 hours at 1400°F.

Smooth and notched tensile and creep rupture tests were carried out on these materials to characterize their mechanical behavior from 1000° to 1400°F (the testing required for the first three conditions was more limited since they had already been evaluated at 1000° and 1200°F in the initial stage of the program). Extension of test temperatures to 1400°F was incorporated into the study to help delineate the cause of the notch sensitivity by lowering the creep resistance, thus shifting the strength-controlling property to a greater extent from tensile to creep characteristics. In addition, the material solution treated at 1825°F was selected for testing at 950° and 900°F since it exhibited the most severe time-dependent notch sensitivity at 1000°F. This heat treated material was the most suitable to enable determination of whether or not the time-dependent notch sensitive behavior could be expected at temperatures below 1000°F.

The microstructures of these heat treated materials were examined in detail. Particular emphasis was placed on the establishment of the dislocation mechanisms operative during the smooth specimen test exposures which necessitated the use of transmission

electron microscopy.

### Notched Specimen Geometry

As part of the research, the testing of sheets of variable thickness was carried out to further delineate the influence of specimen geometry on the time-dependent notch sensitivity. It was desirable to determine to what extent the extensive characteristics developed for the 0.026-inch thick materials are applicable to other sheet thicknesses. Initially tensile and creep-rupture tests at 1000° to 1400°F were carried out on 0.050-inch thick cold worked Waspaloy sheet heat treated 1/2 hour at 1975°F plus 16 hours at 1400°F. Comparison of these results were made with those obtained for the 0.026-inch thick material in the same heat treated condition. As these materials were from different heats, experimental variables other than thickness could have biased the results. Consequently, the following materials were tested after heat treatment 1/2 hour at 1975°F plus 16 hours at 1400°F:

- (1) 0.050-inch thick Waspaloy mechanically thinned to 0.026-inch prior to heat treatment, machining of edge-notched specimens and creep-rupture testing at 1000°, 1100° and 1200°F.
- (2) 0.310-inch thick Waspaloy plate hot rolled, subsequently cold reduced 25 - 30 percent to sheets ranging in thickness from 0.013 to 0.075 inches and then heat treated. Selected testing of smooth specimens at 1000° to 1400°F and sharp-edged notched specimens at 1000°, 1100° and 1200°F was used to characterize

the creep-rupture characteristics as a function of thickness.

### Significance of "Yielding"

The relaxation of high stresses introduced by the presence of notches was expected to be a major factor influencing the notch sensitivity. Relaxation due to "yielding" (time-independent plastic deformation) occurs on loading. Further relaxation can occur by subsequent creep (time-dependent plastic deformation). As a means of increasing the relaxation by "yielding", notched specimens (annealed and aged 16 hours at 1400°F) were loaded to a relatively high stress at 1000°F (115 ksi) and, subsequently, rupture tested at 1000° to 1400°F at lower stress levels. Similarly, in order to determine the effect of "yielding" on smooth specimen mechanical properties, specimens were plastically strained small amounts at temperatures from 1000° to 1400°F prior to creep-rupture testing at these temperatures.

## EXPERIMENTAL PROCEDURES

### Reduction of 0.310-inch Plate and 0.050-inch Thick Sheet

Sheets of varying thickness were obtained by rolling and machining as-received materials. Longitudinal rectangular blanks of the 0.310-inch thick plate (W-III) were hot rolled with reheats at 2000°F between passes, pickled, and subsequently cold reduced to the final thicknesses: 0.013, 0.020, 0.026, 0.038, 0.050, 0.062 and 0.075 inches. The rolling schedules were designed so that the amount of cold reduction was maintained at 25 - 30 percent (in consequence the amount of hot reduction varied according to the final thickness).

Longitudinal blanks of 0.05-inch thick material (W-II) were reduced in thickness to 0.026 inches by machining on both sides in a lathe. This reduction was confined to the center section of the blank which would encompass the parallel reduced section of notched specimens machined from the blanks.

### Heat Treatment

Longitudinal rectangular blanks were cut from all of the sheet materials for heat treatment. For the 0.026-inch thick material (W-IV) blanks were also cut in the transverse direction.

The blanks were solution treated individually in an argon atmosphere. Subsequent air-cooling was rapid enough to limit surface oxidation and sufficiently suppress  $\gamma'$  precipitation so that the particle size could be controlled by the aging treatment.

Blanks for subsequent use as test specimens were aged in air and air cooled. To prevent warping during these treatments, they were clamped in a fixture in batches of 10 or 12. One half inch squares of solution treated blanks of the 0.026-inch thick material (W-I) were aged individually in air for hardness testing.

### Test Specimens

#### Smooth Specimens:

The smooth specimens (fig. 2) were prepared by milling bundles of the rectangular blanks. Approximately ten specimens were machined at one time by use of a clamping fixture which enabled accurate dimensional control.

#### Notched Specimens:

The sharp edge-notched specimens had an elastic stress concentration factor,  $K_t > 20$  (fig. 2). As was the case for the smooth specimens, ten blanks were machined at one time using a fixture. The reduced section of the specimen was first milled to size. The notches were then ground almost to size using an alundum wheel having a 60-degree included angle. The final radii of the sharp notches were obtained by drawing a sharp carbide tool through the notches until the limits of the dimensions were obtained as measured with a 50X optical comparator.

## Testing Methods

### Hardness

Diamond pyramid hardness (DPM) tests were used to measure the response of solution treated materials to a range of aging treatments. Samples of the 0.026-inch thick heat treated materials were metallographically polished to the approximate mid-thickness. Five impressions were made on each sample using a 100 kilogram load. Both diagonals of each impression were measured. A statistical analysis of this technique had shown that in the range of 300-340 DPH, a hardness difference of 7 DPH was significant. This range of significance increased to 9 DPH in the range from 340-400 DPH (ref. 4).

### Tensile Tests

All of the tensile tests were conducted using a hydraulic tensile machine. Smooth specimens were tested at a cross head speed of approximately 0.01 inches per inch per minute up to about 2 percent deformation. The strain rate was then increased to about 0.05 inches per inch per minute until failure. Notched specimens were loaded at a rate of 1000 psi per second.

Heating was provided by a resistance furnace surrounding the specimen, strain gauge, and holder assembly. The furnace was first heated to within 50°F of the desired temperature. The specimen was then placed in the hot furnace and in a period of not more than four hours brought up to the test temperature and an acceptable temperature distribution along the specimen attained. The temperature of the



smooth specimens were measured by three thermocouples, one at each end and one at the center of the gauge section. Only a single center thermocouple was used for the notched specimens. All of the thermocouples were shielded from direct radiation. (ASTM-E139, recommended practices were followed in the control of test temperature and distribution).

The smooth specimen tests were instrumented to measure strain by means of a modified Martens optical extensometer system. Pairs of extensometer bars were attached to collars clamped to the gauge section of the specimens (2-inch gauge length). The stems of mirror assemblies were placed between these pairs. These reflected an illuminated scale located about five feet in front of the test unit. The differential movements of the top and bottom pairs of bars caused by the elongation of the specimen resulted in a rotation of the mirrors. The changes in scale numbers reflected by the mirrors were observed through a telescope mounted next to the illuminated scale. The optical lever magnified these changes and converted them into a large change in the reflected scale reading. This system detected specimen strains as small as 10-millionths of an inch.

#### Creep-Rupture Tests

The creep-rupture tests were conducted in creep testing machines in which the stress was applied through a lever system having a lever to arm ratio of about 10 to 1. The specimens were gripped by means of pins which passed through each end of the

specimen and into holders fitted to a universal joint-type assembly to promote uniaxial loading. The heating and temperature control of the specimens was the same as previously described for the tensile tests.

Strains for the smooth specimens were measured by means of the extensometer system described for tensile testing. Strain measurements were recorded as each weight was applied during loading and periodically throughout the test. When rupture occurred, an automatic timer, activated by the fall of the specimen holder, registered the test time to the nearest one-tenth of an hour.

### Structural Examination

#### Optical Microscopy

The structures were examined after polishing down from the flat surfaces of the sheets rather than through the thickness cross sections. To accomplish this the samples for examination were mounted in plastic and wet ground on a rotating lap through a series of silicon carbide papers, finishing at 600-mesh grit. Final polishing was carried out on a cloth-covered rotating lap using fine diamond compound and then on a vibratory polisher in an aqueous media of Linde "B" polishing compound.

The specimens were etched electrolytically in "G" etch, an etchant developed by Bigelow, Amy and Brockway (ref. 5). Etching was conducted at 1-1/2 volts and a current density of approximately 0.2 amperes per square inch for a period of 10-20 seconds. The

composition of the etchant is as follows:-

"G" Etch

$\text{H}_3\text{PO}_4$  (85%)    12 parts

$\text{H}_2\text{SO}_4$  (96%)    47 parts

$\text{HNO}_3$  (70%)    41 parts

Standard techniques of optical examination and photomicrography were employed.

Electron Microscopy

The materials were studied by both replica and transmission techniques using a JEM-6A electron microscope.

Collodion replicas were prepared from polished surfaces of the as-heat treated materials etched with "G" etch. The replicas, mounted on copper grids, were shadowed with chromium to increase the contrast before they were examined in the electron microscope operated at 80 KV.

A study by transmission electron microscopy was made of thinned samples cut from the as-heat treated materials and tested smooth specimens. In the latter case the samples were from the gauge section somewhat removed from the fracture. Before thinning, the samples measured approximately 0.5-inch square by 0.026-inch thick. While held on a flat metal block by wax, these were reduced in thickness to approximately 0.005-inch by grinding both sides on wet silicon carbide papers finishing with 600 grit. After removal of the wax by acetone, further reduction in thickness was achieved by electropolishing using the Bollman technique (ref. 6).

The electrolyte used was a chilled mixture of 6 percent perchloric acid and 94 percent acetic acid. Electropolishing was carried out at an applied voltage of 38 volts and a current density of about 0.5 amps/cm<sup>2</sup>. Precipitated phases at the grain boundaries and the grains themselves thinned at approximately the same rate permitting study of all the microstructural features of the alloy. An insulating lacquer (Microstop) was applied to the edges of the samples to prevent preferential dissolution in these areas during electropolishing. By varying the relative position of the specimen and the pointed electrodes used, it was possible to control the amount of metal removed from various areas of the specimen. This technique was particularly useful in eliminating the slightly rounded surface produced by the mechanical polishing. Electropolishing was continued until a hole developed in the specimen, at which stage the lacquer was dissolved in acetone. The specimen edges, including those of the hole, were then relacquered and the process continued until the film thickness was less than 0.002 inch.

Final thinning by electropolishing before microscopic examination was accomplished by the Dupres Method (ref. 7). This technique involved welding a small section (approximately 0.2 inches square) of the thinned metal between stainless steel washers; these washers not only supported the film, but also produced a uniform current density during the final electropolishing. Polishing was discontinued as soon as a hole developed in the film. The film was then studied and photographed in the electron microscope operated at 100 KV.

## X-Ray Diffraction

X-ray diffraction analysis of extracted residues was used to identify the carbides present in the as-heat treated materials. The residues were obtained by preferentially dissolving the matrix in a bromine-alcohol solution and centrifuging the resulting suspensions. The residues were washed repeatedly with alcohol until the supernatant liquid was clear. The extracts were then dried and formed into thin wires using a Duco Cement binder. X-ray exposures were conducted in a 144.6 mm diameter Debye camera using nickel-filtered copper radiation (40 KV, 16mA) for a period of four hours. The line positions were measured and the "d" values calculated. The results were then analyzed by comparison with standard patterns available from the literature, and in particular, from the files of The Joint Committee on Powder Diffraction Standards.

## INFLUENCE OF HEAT TREATMENT ON THE MECHANICAL CHARACTERISTICS

Initially, exploratory tensile and creep-rupture tests were carried out at 1000° and 1200°F for eight different heat treatments of 0.026-inch thick sheet (W-I). Based on these results together with those from hardness and metallographic studies, three of these heat treated conditions were selected for in depth evaluation. Three additional heat treatment variations were also selected for this intensive evaluation at test temperatures from 1000° to 1400°F.

### Exploratory Tests At 1000° And 1200°F

Tensile tests at 1000°F and creep-rupture tests at 1000° and 1200°F were carried out for the following eight heat treated conditions:

<u>Solution Treatment</u>		<u>Aging Treatment</u>
1. 1/2 hour at 1825°F	+	16 hours at 1400°F
2. 1/2 hour at 1825°F	+	10 hours at 1700°F
3. 1/2 hour at 1900°F	+	16 hours at 1400°F
4. 1/2 hour at 1900°F	+	10 hours at 1700°F
5. 1/2 hour at 1975°F	+	16 hours at 1400°F
6. 1/2 hour at 1975°F	+	10 hours at 1700°F
7. 1/2 hour at 2150°F	+	16 hours at 1400°F
8. 1/2 hour at 2150°F	+	10 hours at 1700°F

The heat treatment variations studied resulted in a wide range of mechanical characteristics (tables 1 through 3, figs. 3 through 11). The principal features, particularly those related to the nature of the

time-dependent notch sensitivity were as follows:

- (1) The materials aged 16 hours at 1400°F exhibited time-dependent notch sensitivity. At the shorter test times the notched specimen rupture curves at 1000°F were somewhat below those for smooth specimens (figs. 3 through 6). However, the notched to smooth specimen rupture strength ratios (N/S) were of the same order as determined by tensile tests (table 3). At time periods varying from 200 to 3000 hours the notched specimen rupture curves exhibited breaks so that the strengths fell rapidly with time. There was no evidence that the curves for smooth specimens exhibited similar behavior. Consequently, at the longer test times beyond the breaks, the notched to smooth rupture strength ratios decreased with increasing times to values considerably below those obtained in tensile tests, i. e., the materials exhibited time-dependent notch sensitivity. Analysis of the data for these materials showed that the time-dependent notch sensitivity occurred when the notched specimens were loaded (based on the net section at the base of the notch) below the approximate 0.2 percent offset yield strength as determined in the tests of smooth specimens. The time at which the break occurred was significantly shorter for solution treatment at 1825°F than for the materials solution treated at the higher temperatures (figs. 3 through 6). There was some indication that increasing the solution temperature from 1900° to 2150°F increased the time at which change in slope of the rupture

curves occurred for notched specimens.

Time-dependent notch sensitivity also occurred at 1200°F for the materials aged at 1400°F after solution treatment at 1900°, 1975° and 2150°F (figs. 4, 5, 6). At relatively short times the N/S rupture strength ratios fell to very low values (table 3). At longer times the rupture curves were less steep indicating a decrease in notch sensitivity. The rupture curve at 1200°F for notched specimens for the materials solution treated at 1825°F and aged at 1400°F was approximately parallel to that for smooth specimens (fig. 3); the notched to smooth rupture strength ratios (about 0.75) were similar to the tensile strength ratio at 1200°F (0.78). Thus, for this material increasing the temperature from 1000°F to 1200°F resulted in elimination of the time-dependent notch sensitivity.

- (2) In marked contrast to the materials aged at 1400°F, for those aged at 1700°F no time-dependent notch sensitivity was observed at 1000° or 1200°F (figs. 3 through 6). The notched to smooth rupture strength ratios were generally higher than those obtained in tensile tests (table 3).
- (3) The heat treatment variations resulted in a wide range of smooth specimen mechanical characteristics (figs. 7 through 11). In the tests at 1000°F the notched and smooth tensile strengths, the smooth specimen rupture strengths, and the creep resistance decreased with increasing temperature of the solution treatment (table 1, figs. 7, 9). For a given solution treatment, aging at



1400°F resulted in higher values (except for the rupture strengths of the materials solution treated at 2150°F) than for aging at 1700°F. Smooth specimen rupture strengths and creep resistances at 1200 °F (with the exception of the material solution treated at 2150°F and aged at 1400°F) were, particularly at the lower stresses, much less dependent on heat treatment than at 1000°F (figs. 7,9).

The elongations at rupture for tests at 1000° and 1200°F were higher for aging at 1700°F than for aging at 1400°F (table 2, fig. 10). The elongation for the material heat treated at 2150°F and aged at 1700°F fell to a very low level at the longer test times at 1200°F. For aging at 1400°F (fig. 10) most striking was the trough, or minimum elongation, exhibited at 1200°F by the materials heat treated at 1825°F and 1975°F (and presumably 1900°F). For the material solution treated at 2150°F and aged at 1400°F, the elongations fell to low values.

The reduction in area values reported (table 2, fig. 11) are the average values across the fractures. Due to the complex nature of the fracture (discussed in a subsequent section) considerable variations in reduction of area occurred along the path of the fracture. The values measured were relatively high and showed less variation with heat treatment than was the case for the rupture elongations.

Consequently, for each solution treatment increasing the aging temperature from 1400° to 1700°F, resulted in elimination of the

time-dependent notch sensitivity. This was also accompanied by clearly definable changes in the smooth specimen characteristics. However, when the results from all of the heat treated conditions were considered, no correlation was evident between the notch sensitive behavior and any particular individual characteristic.

### Selected Heat Treated Conditions

Based on the exploratory rupture test results and on the subsequently reported hardness tests and metallographic studies, the following three heat treatments were selected for intensive study.

<u>Solution Treatment</u>		<u>Aging Treatment</u>
1.	1/2 hour at 1825°F +	16 hours at 1400°F
2.	1/2 hour at 1975°F +	16 hours at 1400°F
3.	1/2 hour at 1975°F +	10 hours at 1700°F

Also based on the results of initial studies, the following three additional heat treatments were incorporated into the investigation:

<u>Solution Treatment</u>		<u>Aging Treatment</u>
1.	1/2 hour at 1975°F +	24 hours at 1550°F
2.	1/2 hour at 1975°F +	24 hours at 1550° plus 16 hours at 1400°F
3.	4 hours at 1975°F +	24 hours at 1550°F plus 16 hours at 1400°F

Tensile and creep-rupture tests from 1000° to 1400°F were carried out for both smooth and sharp-edge notched specimens for these heat treated materials. For the material solution treated at

1825°F, the creep-rupture testing was extended to include 900° and 950°F.

### Tensile and Creep-Rupture Strength Characteristics

From analysis of the added experimental data (tables 4 through 10, figs. 12 through 16) the observations derived from the exploratory test results were further clarified and extended.

- (1) For the materials solution treated 1/2 hour at 1825°F and 1975°F and aged 16 hours at 1400°F, the notched specimen rupture curves showed three regimes of behavior: (a) At the lower test temperatures the steepness of the curves increased drastically at varying time periods. (b) At intermediate temperature levels an upward break was observed. (c) At the highest test temperatures utilized, the curves did not show any marked slope changes (figs. 12, 13, 16). Since the smooth specimen rupture curves did not exhibit similar changes, the increases in steepness of the curves at the lower test temperatures resulted in time-dependent notch sensitivity. The notched to smooth rupture strength ratios at these test temperatures fell to values considerably below those obtained in tensile tests (tables 5, 6). With increasing test temperature and longer rupture times, the rupture strength ratios increased until, at the highest temperatures values of about 1.0 were obtained, i. e., the time-dependent notch sensitivity decreased. It was also evident that the time-dependent notch sensitivity occurred at somewhat longer times and higher temperatures for the material

solution treated at 1975°F than that heat treated at 1825°F.

- (2) For the material solution treated 1/2 hour at 1975°F: Increasing the aging temperature from 1400°F to 1700°F resulted in elimination of the time-dependent notch sensitivity (table 6, 7; figs. 13, 14, 16). It also resulted in lower strength levels. However, after aging 24 hours at 1550°F no time-dependent notch sensitivity was observed, and the strength levels remained high (table 8; figs. 15, 16). No significant change in smooth and notched specimen strengths resulted from the addition of a second lower temperature aging treatment at 1400°F, i. e., for the multiple aged materials, 1/2 hour at 1975°F plus 24 hours at 1550°F plus 16 hours at 1400°F and 4 hours at 1975°F plus 24 hours at 1550°F plus 16 hours at 1400°F (tables 9, 10; fig. 15).
- (3) At the lower test temperatures, the heat treatments studied resulted in considerable ranges of smooth and notched specimen creep-rupture strengths (fig. 16). At the higher temperatures and longer test times the strengths became more similar independent of specimen type or heat treatment. The results indicated that this similarity of rupture strengths occurred at times greater than about 10,000 hours at 1300°F and 500 hours at 1400°F (fig. 16). These factors were particularly evident from analysis of the rupture data in terms of time-temperature effects, the results of which are included as Appendix I. Metallographic studies reported in a subsequent section indicated that the similarity of strengths at

the higher temperatures and times resulted, at least in part, from gamma prime growth during the test exposures.

- (4) Rupture tests at 900° and 950°F for the material heat treated 1/2 hour at 1825°F plus 16 hours at 1400°F demonstrated that the time-dependent notch sensitivity can occur at temperatures below 1000°F (table 5; fig. 12). The occurrence of notch sensitivity at low temperatures is considered in greater detail in Appendix I.

#### Ductility in Creep-Rupture Tests

The elongation at rupture varied considerably with the test conditions (temperature and stress) as well as heat treatment (tables 5 through 10; fig. 17). For the materials which exhibited time-dependent notch sensitivity (materials solution treated at 1825°F and 1975°F and aged at 1400°F) an apparent correlation was evident between the occurrence of time-dependent notch sensitivity and elongation at rupture. The elongation values decreased with increasing rupture time for the tests at the lower temperatures which could be correlated with increasing time-dependent notch sensitivity. At higher test temperatures, the rupture elongations increased with time, which corresponded to decreasing notch sensitivity. However, from analysis of the data for the other heat treatments, it was evident that no clearly definable correlation existed between the notch sensitivity and elongation at rupture that was applicable to all heat treatments and test conditions. For example, the elongations for the materials heat treated 4 hours at 1975°F plus 24 hours at 1550°F plus 16 hours at 1400°F and 1/2 hour

at 2150°F plus 10 hours at 1700°F fell to very low levels at 1200°F (2 and 2.2 percent respectively) and yet no time-dependent notch sensitivity was observed (figs. 17, 10).

The reduction in areas (tables 5 through 10) showed less variation than was the case for the elongations. The values decreased with increasing rupture time at the lower test temperatures (1000° and 1100°F), and remained at relatively low levels at the intermediate temperatures (1200°F and 1300°F), while higher values occurred at the highest temperatures (1400°F). Within this general scheme, small deviations in behavior occurred depending on heat treatment. Again, there was no clearly definable correlation between the reduction in area and the occurrence of time-dependent notch sensitivity.

Thus, from the data available it was not possible to establish a correlation between rupture ductility and the time-dependent notch sensitive behavior. Analysis of the deformation-time characteristics, reported in a subsequent section, demonstrates that factors that contribute to the ductility rather than the ductility per se, control the notch sensitivity. Combinations of the controlling factors may occur so that a correlation occurs between low ductility and notch sensitivity, and high ductility and no time-dependent notch sensitivity. It is equally possible however, that high rupture ductility could be accompanied by the occurrence of time-dependent notch sensitivity or relatively low ductility by no notch sensitivity.

## FRACTURE CHARACTERISTICS

Examination of both notched and smooth failed specimens showed that, for all but a few tensile tests, the fractures consisted of two distinct parts (fig. 18). One section was perpendicular to the loading axis and tended to be dark in color from oxidation. The remainder of the fracture across the specimen was not appreciably discolored by oxidation and was a typical shear failure, slanted through the thickness. Metallographic examination showed that the above fractures were intergranular and transgranular respectively. A series of micrographs taken at regular intervals along the fracture of a ruptured notched specimen are presented in Figure 19 (because of its large grain size, the material solution treated at 2150°F and aged at 1400°F provided the clearest illustration at low magnification of the fractures that occurred in a similar manner for all of the heat treated materials evaluated). In these micrographs the presence of the intergranular and transgranular fractures are clearly discernible. Intergranular cracks were also found in several of the notched specimens that were discontinued before rupture, particularly those which had been exposed for nearly their expected rupture time (fig. 20). Some of the specimens which failed at the pin holes (used to attach the specimens to the holders for testing) also had intergranular cracks at the notches (fig. 21). In no case were transgranular cracks found in specimens not tested to rupture. These observations indicated that failure of the specimens occurred by initiation and

relatively slow growth of the intergranular crack followed by shear failure in a time period short enough to avoid discoloration by oxidation.

Measurements across the specimens at the fractures showed that the reduction in thickness was greater in the regions of the transgranular crack than at the intergranular fracture. The microstructures also showed evidence of deformation associated with the transgranular fracture (fig. 19). The transgranular failures were therefore ductile. The data for the reductions in area included in the tables were calculated using the average values for the reduction in thickness across the fractures.

In order to provide an understanding of the significance of the intergranular and transgranular cracking to the mechanical properties, in particular the time-dependent notch sensitivity, the two following features were evaluated: (i) measurements of the lengths of the intergranular part of the fractures, and (ii) intergranular crack initiation and growth rates for smooth and notched specimens. The following features were evident:

- (1) The lengths of the intergranular cracks (expressed as percentage of the specimen widths) increased with decreasing test stress, and thus, with increasing rupture times (tables 2, 3, 5 through 10; figs. 22 through 25). This result was to be expected since, at the lower test stresses, the intergranular cracks have to grow more before the stress becomes high enough to initiate the transgranular fracture.



The presence of intergranular cracks in the smooth specimens reduced the transgranular strengths. For example, the material heat treated 1/2 hour at 1975°F plus 16 hours at 1400°F and tested at 80 ksi at 1200°F fractured with 34 percent of the width intergranular. Consequently, the "transgranular strength" was  $80 \times 100/66 = 120$  ksi. This value was less than the transgranular strength derived from the smooth specimen tensile test at 1200°F (168 ksi) but similar to that from the notched tensile test (124 ksi). Thus, once an intergranular crack was formed in a smooth specimen, its behavior became somewhat similar to that of a notched specimen. For notched specimens, the presence of the intergranular crack had relatively little influence on the transgranular strength, i. e., the notch "simulated" the presence of a crack. If in fact, smooth and notched specimens for given test conditions did behave exactly the same once intergranular crack initiation had occurred then it would be expected that they would exhibit the same intergranular crack lengths at fracture (expressed as a percentage of the width). Although this was approximated under some test conditions, it was generally not the case (figs. 22, 23). The differences in intergranular crack lengths at each stress level probably resulted principally from differences in the specimen geometries and variations in the distribution of the creep damage in the two specimen types. This latter factor is discussed subsequently. The influence of the transgranular strength variation due to heat

treatment on the length of intergranular cracks was most clearly evident in the data from smooth specimens (fig. 24). For a given test stress, the intergranular crack length increased with the transgranular strength (as reflected by tensile tests). The intergranular crack growth rates increased as cracking progressed due to the increase in stress on the load bearing section (fig. 26). Consequently, although the transgranular strength influenced the intergranular crack length in a ruptured specimen it could be expected to have only a limited effect on the rupture time and hence on the time-dependent notch sensitivity. However, microstructural features such as the  $\gamma'$  size which influence the transgranular strength also can be expected to affect the initiation and growth of intergranular cracks and, therefore, be reflected in rupture times and variations in notch sensitivity.

- (2) The deformation that occurred prior to intergranular crack initiation in the smooth specimen creep-rupture tests, was relatively uniform through the gauge section. Because of this, the major intergranular crack (that which led to ultimate failure) initiated randomly in the specimens, sometimes at the edges and at the center of others. In addition when extensive subsidiary cracks were evident, they were observed throughout the gauge section (figs. 27, 28).

Limited test results demonstrated that visible intergranular cracks formed very late in the rupture life of the smooth specimens. Samples of the material solution treated 1/2 hour at 1975°F and aged

16 hours at 1400°F were periodically cooled under load, examined for cracks and reheated under load to the test temperature. For specimens exposed at 1000°F under 130 ksi (ruptured in 734 hours) and at 1400°F under 38 ksi (ruptured in 971 hours) no cracks were detected by visual examination at 97 and 95 percent of their rupture lives respectively. These observations demonstrate that the crack growth rate was rapid. This would be expected for all heat treated conditions since the introduction of a crack would occur when a high level of creep damage existed and the crack would result in (i) a stress concentration at the crack tip and consequently introduce localized deformation in this area, and (ii) an increase in stress due to the decrease in the load bearing section at the crack.

- (3) Periodic examination of rupture tested notched specimens heat treated 1/2 hour at 1975°F plus 16 hours at 1400°F showed that the presence of the edge-notches resulted in highly localized deformation. In the early stages of the tests, dimples formed at the base of the notches. Metallographic examination showed that creep induced microcracks occurred in these regions (fig. 29a). With continued exposure, intergranular cracks extending through the thickness, became visible at one or both notches (fig. 20, 29b). The life fractions at which visible cracks formed were as follows:-

<u>Test Temp (°F)</u>	<u>Stress (ksi)</u>	<u>Rupture Time (hours)</u>	<u>Life Fraction for Crack Initiation (percent)</u>
1000	105	347	>66
	95	1454	65
	85	2634	80
1200	50	256	>87
1400	50	44	>91
	38	851 ph	60*

> - No visible cracks detected at this time period, no further observations were made prior to rupture.

ph - Pin hole failure.

\* - Estimated using rupture life established from a continuous test at this condition (1100 hours).

Although considerable data scatter was evident in the results, it was concluded that visible cracks formed after at least 60 percent of the rupture life, but at shorter life fraction than for smooth specimens. The crack growth rates increased with time reflecting the increase in stress on the load bearing sections (fig. 26). However, the growth rates were slower than for the smooth specimens. Presumably this resulted because little creep damage occurred ahead of the dimpled region during crack initiation. This was supported by the observation that the dimpled region (the area of severe deformation and extensive microcracking) moved ahead of the crack tip as it progressed across the specimen (fig. 29b).

Consequently, for the material solution treated 1/2 hour at 1975°F and aged 16 hours at 1400°F which exhibited time-dependent

notch sensitivity, notched specimen rupture fractures were initiated by localized deformation at the base of the notches. This resulted (as is discussed in the following section) from the deformation accompanying relaxation of the high stresses introduced by the presence of the edge-notches.

## STRESS RELAXATION

Relaxation of the stress concentrations introduced by the presence of the edge-notches can occur due to "yielding" on loading (time-independent plastic deformation) and by subsequent creep (time-dependent plastic deformation). Experiments and data analysis were carried out in an effort to determine the significance of each of these deformation processes in the occurrence of time-dependent notch sensitivity.

### Relaxation By "Yielding"

In order to study the significance of the contribution of "yielding" to relaxation of stresses in notched specimens, experiments were carried out which were designed to increase the amount of time-independent deformation that occurs on loading in the rupture tests. To accomplish this, notched specimens were preloaded prior to rupture testing to stresses high enough to cause yielding completely across the section at the base of the notch. The study was conducted using material (W-IV) annealed and aged 16 hours at 1400°F. Initially, a number of notched and smooth specimen creep-rupture tests were carried out without preloading (table 11) in order to fill out the data already available from a previous investigation (ref. 1).

Notched specimens were preloaded at 1000° to 115,000 psi prior to rupture testing at temperatures from 1000° to 1400°F. The results (table 12) are compared graphically with those obtained without preloading in Figure 30b. Expressing the data in terms of a time-temperature

parameter (Larson-Miller; see Appendix I) was chosen simply because it was an effective method of presenting the data. It is clearly evident from the results that the preloading of notched specimens prior to rupture testing at lower stress levels reduced, if not eliminated, the time-dependent notch sensitivity. However, from these results it is impossible to separate effects directly related to varying the amount of stress relaxation from other effects which may have been due to the introduction of small amounts of plastic strain into the material.

To provide a basis for the separation of the effects contributing to the notched specimen behavior, limited creep-rupture tests were also conducted on smooth specimens after pre-straining small amounts at the test temperatures (table 12). The results indicated that strains (such as would be expected to occur when the notched specimens were yielded across the section at the base of the notch prior to creep-rupture exposure) had relatively little effect on the smooth specimen rupture life (fig. 30a). Little or no shortening of rupture life was observed at the test temperatures where the time-dependent notch sensitive behavior occurred, i. e., 1000° and 1200°F. In tests at 1400°F, where the material did not exhibit time-dependent notch sensitivity, the rupture life was shortened to a limited extent by pre-straining.

In view of the above results, it was apparent that the relaxation of the stress concentrations by yielding at the base of the notches had a very marked influence on the time-dependent notch sensitivity. It should also be noted that no time-dependent notch sensitivity was

observed in tests above the approximate 0.2 percent offset yield stress. In these cases, subsequent to "yielding" on loading, the stresses across the specimens at the base of the notches would have approximated the nominal stresses. At loading stresses below the yield stress, the stress concentrations are not fully relaxed by "yielding". The stresses at the base of the notches remain at relatively high levels (at about the yield strength) until they are relaxed by subsequent creep. Thus, the occurrence of the time-dependent notch sensitivity and its dependence on heat treatment must be related to the manner by which the materials relax stress concentrations by creep.

#### Relaxation By Creep

In rupture tests of notched specimens loaded to nominal stresses below the yield stress, creep deformation must occur at the base of the notches in order to reduce the stress level in these regions to the nominal. Premature failure of notched specimens, i. e., time-dependent notch sensitivity, will occur if all or a large proportion of the rupture life of the material is used up in the relaxation of the stresses. This can occur since small increases in stress result in considerable shortening of rupture life. Thus, whether or not a material exhibits time-dependent notch sensitivity will be dependent on the ability of the material to deform small amounts by creep without causing excessive damage, i. e., without using up considerable rupture life.

The creep behavior of the smooth specimens was examined to



determine whether their characteristics could be correlated with the occurrence of the time-dependent notch sensitivity. Data from the creep deformation-time curves were used to construct iso-creep strain curves for each test temperature on plots of life fraction versus test stress. The life fraction, which is a convenient method of expressing the rupture life utilized for any given exposure is defined as:-

$$\frac{\text{time at the given stress}}{\text{rupture time at the same stress}}$$

These values were calculated for a number of levels of creep strain for each test. For the majority of the notched tests creep deformation of about 0.1 or 0.2 percent would ensure relaxation of the elastic stresses from the approximate yield stresses to the nominal stresses. Iso-creep strain curves were derived for these levels of creep strain and also for 0.5, 1 and 2 percent. These latter curves helped establish with greater certainty those for 0.1 and 0.2 percent creep strain. Extrapolations of the curves were drawn by eye with consideration of the shapes of the curves within the range of test stresses and the relationships between adjacent curves.

It was evident from the results (figs. 31 through 34) that the variations in the time-dependent notch sensitivity behavior with heat treatment and test temperature (and time) could be correlated with the characteristics of the iso-creep strain curves.

#### Materials Exhibiting Time-Dependent Notch Sensitivity

For the material heat treated 1/2 hour at 1825°F and aged 16

hours at 1400°F, the results for test temperatures of 950°, 1000° and 1100°F showed that the life fractions for small amounts of creep strain increased drastically as the test stress decreased (fig. 31). For notched specimens loaded to nominal stresses somewhat below the yield strength the relaxation of the stresses (from the approximate yield stress) to the nominal would utilize considerable, if not all, of the creep-rupture life of the material at the base of the notch. Thus at these test temperatures time-dependent notch sensitivity would be expected to occur, i. e., for notched tests at nominal stresses below the yield stress and where the life fractions were high for 0.1 or 0.2 percent creep strain (below approximately 155,000 psi (yield stress) at 950°, 130,000 psi at 1000° and 100,000 psi at 1100°F). At test temperatures 1200°, 1300° and 1400°F, the life fractions for 0.1 or 0.2 percent creep strain were low for all stress levels. Consequently, little or no time-dependent notch sensitivity would be expected at these temperatures. The above predictions are in agreement with the time-dependent notch sensitive behavior observed (fig. 12).

Similar relationships between the nature of the iso-creep strain curves and the notch sensitive behavior were evident for the material solution treated 1/2 hour at 1975°F and aged 16 hours at 1400°F (fig. 32). For test temperatures of 1000° and 1100°F the life fractions utilized for the given small amounts of creep strain increased markedly with decreasing stress. As was observed experimentally, time-dependent notch sensitivity would be expected for notched specimens tested below

about 115,000 psi at these temperatures (fig. 13). At 1200° and 1300°F as the test stress decreased the life fractions for 0.1 and 0.2 percent strain increased to relatively high levels and then subsequently decreased. These results are consistent with behavior observed, i.e., an increase and a subsequent decrease in notch sensitivity with time at these temperatures. At 1400°F the life fractions were at relatively low levels and no time-dependent notch sensitivity was observed. These results and the others reported subsequently were obtained from the study of 0.026-inch thick materials. A study of the influence of sheet thickness for materials solution treated 1/2 hour at 1975°F and aged 16 hours at 1400°F showed that the time-dependent notch sensitivity behavior was independent of thickness for the range studied, 0.013 to 0.075-inch (Appendix II). Consequently the correlation is applicable at least for this range of sheet thickness.

The creep data available for other heat treated materials which exhibited time-dependent notch sensitivity (figs. 4, 6) were too limited to develop iso-creep strain curves such as presented above. Consequently the iso-creep data have not been presented. Analysis of the data, however, showed that the results for these materials are not inconsistent with the above concepts.

#### Materials Which Did Not Exhibit Time-Dependent Notch Sensitivity

Within the range of the test data available for the material solution treated 1/2 hour at 1975°F and aged 10 hours at 1700°F, the life fractions for 0.1 and 0.2 percent creep strain remained at relatively

low levels independent of stress at test temperatures from 1000° to 1400°F (fig. 33). Within the times of the notched specimen tests, no time-dependent notch sensitivity was observed (fig. 14). This is consistent with the iso-creep strain characteristics. However, the iso-creep strain data at 1000° and 1100°F do not indicate whether or not the life fractions increase to high values at very low stress levels. If, in accordance with the trends at the higher temperatures, the slopes of the iso-creep strain curves decrease at the lower stresses, then no time-dependent notch sensitivity would be expected to occur. If, on the other hand, the slopes of the curves continue to increase with decreasing stress, then time-dependent notch sensitivity would be expected to occur at the low temperatures. If this were so, an increase in notch sensitivity would occur at very low notched specimen stress levels, i. e., extremely long times. In fact, rupture tests at 1000° or 1100°F would have to be extremely prolonged (100,000 hours or longer) to demonstrate if the effect exists.

For the materials aged 10 hours at 1700°F after solution treatment at 1825°, 1900° and 2150°F no time-dependent notch sensitivity occurred (figs. 3, 4, 6). Analysis of the available creep data did not reveal inconsistencies from the trends established for the material solution treated at 1975°F and aged at 1700°F.

The iso-creep strain characteristics were also studied for the materials in the following heat treated conditions:

- (a) 1/2 hour at 1975°F and aged 24 hours at 1550°F

(b) 1/2 hour at 1975°F and aged 24 hours at 1550°F plus 16 hours at 1400°F

(c) 4 hours at 1975°F and aged 24 hours at 1550°F plus 16 hours at 1400°F

These three materials exhibited smooth specimen tensile and rupture properties similar to those for the material heat treated 1/2 hour at 1975°F and aged 16 hours at 1400°F (table 4; figs. 15, 16). In contrast to the latter material, the results did not show the occurrence of time-dependent notch sensitivity. Therefore, comparisons were made of the iso-creep strain characteristics of these three materials and those for the material aged at 1400°F (fig. 32). Figure 34 shows the results for the material heat treated 4 hours at 1975°F and aged at 1550°F and at 1400°F. (This particular material was selected for presentation since it was the most extensively tested and of the three heat treated materials it exhibited the lowest rupture ductility). At the higher stress levels, the life fractions for small amounts of strain were considerably less for the material aged at 1550°F and at 1400°F than for the material aged at 1400°F. Therefore, the latter material had a greater tendency for time-dependent notch sensitive behavior. At the lower stresses (higher test temperatures) the life fractions for 0.1 and 0.2 percent strain for both materials were at low levels, and as would be expected no time-dependent notch sensitivity was observed. However, for the material aged at 1550°F and at 1400°F the possibility cannot be excluded that time-dependent notch sensitivity could occur at very prolonged time periods at the lower test temperatures (as discussed for the

material aged at 1700°F).

Consequences Of The Correlation Between Deformation Characteristics  
And The Time-Dependent Notch Sensitivity.

The existence of the qualitative relationship between the nature of the smooth specimen deformation characteristics and the occurrence of the time-dependent notch sensitivity has two ramifications:-

- (1) It can be concluded that there is nothing inherently different about notched and smooth specimen behavior. For example, there are no thermally induced metallurgical effects occurring in the notched specimens that do not also occur in the smooth specimens. Consequently, it is conceivable that mathematical models could be developed to derive notched specimen rupture behavior from the smooth specimen deformation and rupture characteristics. Considerable effort by many researchers has been directed at the derivation of such methods to correlate tensile properties. However no good methods are available for correlation of creep-rupture behavior. This probably reflects the complexities involved. The influence of a great number of factors would have to be integrated into the analysis: calculations of stress and strain states and their variation with time, criteria for deformation and fracture under complex stresses, structural instabilities, etc. Thus, although feasible, the mathematical derivation of notched specimen rupture properties from smooth behavior would be extremely difficult.

- (2) The correlation offers a qualitative basis or technique for providing an indication of whether or not other alloys may be susceptible to the same type of time-dependent notch sensitivity. This is particularly attractive since smooth specimen deformation characteristics are far more readily available than edge-notched sheet specimen rupture data.

## MICROSTRUCTURAL FEATURES CONTRIBUTING TO TIME-DEPENDENT NOTCH SENSITIVITY

Time-dependent notch sensitivity was shown to occur when (i) the notched specimens were loaded below the approximate 0.2 percent smooth specimen offset yield strength; and (ii) test data from smooth specimens indicated that small amounts of creep strain used up large fractions of creep-rupture life. No reasons have been found to suggest that this finding will not prove to be generally applicable. Any alloy which uses up large proportions of its creep-rupture life at small strains should be notch weakened when stress concentrations are not reduced by yielding during loading.

For any particular alloy, techniques for varying the yield strength are known. Methods for altering the creep-deformation versus time relationships (hence the rupture life fractions for small creep strains) are not so obvious. Usually factors which raise the yield strength also reduce the deformation during primary creep, and therefore, act to promote notch sensitivity. In any case, primary creep can be expected to be sensitive to the microstructures. Consequently, these were examined to determine whether variations in microstructural features could be related to the observed notch sensitive behavior.

The various heat treatments and structural changes that occurred during testing resulted in a wide range of microstructural features. Considerable variations were observed in the gamma prime size, grain



size and grain boundary precipitates. It might be expected that the  $\gamma'$  phase would have the greatest influence on the tensile characteristics (particularly the yield strength) and the nature of the creep deformation. The grain boundary structure should be important to the accumulation of creep damage and the initiation of intergranular cracks. Consequently, the microstructural features of the as-heat treated materials and of tested specimens (0.026-inch thick material, W-I) were studied with the emphasis on the above microstructural features. Initially optical metallography, replica and transmission electron microscopy, and X-ray diffraction studies were carried out for the materials in the as-heat treated conditions. Subsequently, smooth specimens which had been tensile and creep-rupture tested were studied by transmission electron microscopy. The materials chosen for this detailed examination were those for which extensive tensile and creep-rupture testing had been carried out at temperatures from 1000° to 1400°F. The specimens examined encompassed the range of test conditions, and therefore, had undergone a wide range in amounts of deformation. Samples were also studied from specimens strained in a tensile machine at 1000°F to smaller amounts than necessary for fracture. These specimens were examined since it was not possible to evaluate the dislocation structures present in the specimens tensile tested to failure at 1000°F due to the very extensive deformation that had occurred.

## Interactions Of Dislocations With Gamma Prime

It might be expected that the resistance to dislocation motion would be dependent on the interaction of the dislocations with the  $\gamma'$  particles, solid solution effects and the grain boundary structures. However of these, only variations in the gamma prime size distributions were associated with marked changes in dislocation structures. The following discussion will, therefore, be directed at the nature of the dislocation interactions with the  $\gamma'$  phase.

### Materials Solution Treated At 1900°, 1975° and 2150°F And Aged 16 Hours At 1400°F.

Typical microstructures of the as-heat treated materials are included as Figures 35 and 36. The  $\gamma'$  particles were too small to be readily resolvable in the electron microscope using replica techniques (figs. 36, c, e, g). When studied using transmission electron microscopy, contrast effects due to the presence of the  $\gamma'$  precipitate were observed. The average diameter of these contrast effects was approximately  $75\text{\AA}$ . Diffraction patterns taken from these areas contained super-lattice reflections, of the type (100), (110), etc. which indicated the presence of an ordered precipitate, proof of the presence of  $\gamma'$  particles. The average  $\gamma'$  size was also taken to be about  $75\text{\AA}$ . This assumption was based on the fact that the mismatch between the  $\gamma'$  and the matrix  $\gamma$  is very small for Waspaloy.

When tested specimens of the material heat treated 1/2 hour at

1975°F plus 16 hours at 1400°F were studied by transmission electron microscopy, the following features were observed. It could be expected that similar structures must also have occurred for the materials aged 16 hours at 1400°F after solution treatment at 1900° and 2150°F.

- (1) For the tensile and rupture tests carried out at 1000°F, the stress levels were above the yield strength of the material. Large amounts of deformation (samples taken about 1/2 inch from the fractures) occurred resulting in extremely high dislocation densities (figs. 37, 38). The dislocations were concentrated on widely separated (111) planes. The dislocation motion mechanism that presumably led to these microstructures was more readily discernible in samples of the specimen strained approximately 2 percent at 1000°F (fig. 39). Many superdislocations, i. e., pairs of perfect dislocations, were observed. These, which have been reported to occur in many precipitation hardened alloys, indicate that the moving dislocations sheared the precipitate particles (ref. 8, 9). The superdislocation arises since the first whole dislocation disorders the  $\gamma'$  which increases the energy of the particles until the second reorders them. In addition, the presence of stacking fault ribbons demonstrates that cross slip did not occur and, therefore, the  $\gamma'$  must have been sheared (ref. 8).
- (2) The specimens studied after rupture testing at 1200°F and 1300°F exhibited relatively low dislocation densities (fig. 40) reflecting their low elongations. The majority of the dislocations were

present in pile ups. In a large number of cases the dislocations were dissociated to form stacking fault ribbons.

- (3) In the specimen tensile tested at 1400°F (exposed to stress levels over the yield stress), the dislocations were also concentrated on discrete slip planes (fig. 41). The dislocation density was high. In some cases stacking faults were observed.
- (4) It was evident from the microstructure of the specimen studied after rupture testing at 1400°F (at 38 ksi ruptured in 1007 hours) that  $\gamma'$  growth had occurred during the test (fig. 42). The average size of the particles increased from about 75Å to 900Å. The presence of the large  $\gamma'$  led to a distinctly different dislocation arrangement in the ruptured specimen than for the previously described cases where the  $\gamma'$  particles remained small. The microstructure of the ruptured specimen exhibited a homogeneous distribution of entangled dislocations (fig. 42a). It must be assumed, however, that in the early part of the test when the  $\gamma'$  particles were small, the dislocations sheared the particles. Thus in the initial stages the microstructure would have been similar to those previously presented.

Gamma prime contrast effects associated with coherency (ref. 9) were observed (fig. 42b) demonstrating that at least some of the  $\gamma'$  particles remained coherent even after an overall deformation of 6 percent.

Materials Solution Treated at 1900°, 1975° and 2150°F And Aged 10  
Hours At 1700°F.

Aging at 1700°F resulted in larger  $\gamma'$  particles than those produced by the 1400°F treatment. Optical micrographs (fig. 35d, f, h) exhibited a mottled appearance within the grains due to the large size of the  $\gamma'$  particles. The degree to which the mottling occurred increased with the severity of the etching. Spherical particles with an average size of approximately 1000Å were evident in photomicrographs from electron microscope replica (fig. 36d, f, h) and transmission studies. In thin foils examined by transmission electron microscopy, contrast effects were observed associated with the  $\gamma'$  precipitate particles. This demonstrated their coherent nature.

The microstructures of tested specimens of the material solution treated 1/2 hour at 1975°F and aged 10 hours at 1700°F exhibited the following:-

- (1) The deformation that occurred was extremely "general"; the dislocations were homogeneously distributed (figs. 43 through 47). This result for large  $\gamma'$  particles was in strong contrast to the previously described cases where the  $\gamma'$  particles were small and the deformation was highly localized (figs. 37 through 41). Even though the  $\gamma'$  particles were large enough to be readily resolvable, it was difficult to discern the nature of the interaction of the dislocations with the particles in the cases where extensive deformation

had taken place. In specimens deformed lesser amounts (particularly the specimen strained to a few percent at 1000°F), it was clear that the dislocation motion occurred by a by-pass mechanism. The dislocations bowed between  $\gamma'$  particles (fig. 44b) leaving pinched off dislocation loops (fig. 44a). Gamma prime particles that retained their coherency after 9% overall elongation are evident from the contrast effects in Figure 47b.

At the high strains developed in the specimen tensile tested at 1000°F there was a tendency for the deformation to be localized (fig. 43a). It was not clear from the micrographs, however, whether this effect resulted from a change in dislocation motion mechanism from by-passing to shearing the  $\gamma'$  particles.

- (2) Some  $\gamma'$  growth occurred during the creep-rupture tests. For example, for the test at 1400°F and 38 ksi the average  $\gamma'$  size increased from approximately 1000Å to 1500Å during the 931 hour test. The growth was less the shorter the rupture time and the lower the test temperature. As would be expected, the increases in sizes were less than was the case for similar exposures for the material aged at 1400°F (in an 1007 hour test at 1400°F the  $\gamma'$  size increased from 75Å to 900Å).
- (3) The frequency of microtwins (figs. 45a, 46b) was most striking because it was considerably greater than that observed for the materials aged at 1400°F. Selected area diffraction patterns were streaked in the  $\langle 111 \rangle$  directions which were perpendicular to

the planes of the twins.

Materials Heat Treated 1/2 Hour At 1975°F Plus 24 Hours At 1550°F,  
1/2 Hour At 1975°F Plus 24 Hours At 1550°F Plus 16 Hours At 1400°F,  
And 4 Hours At 1975°F Plus 24 Hours At 1550°F Plus 16 Hours At 1400°F

Aging 24 hours at 1550°F after the high temperature solution treatment resulted in  $\gamma'$  particles approximately 600Å in diameter, a particle size intermediate between those obtained by aging 16 hours at 1400°F (75Å) and aging 10 hours at 1700°F (1000Å). For the multiple aging treatment, besides precipitation at 1550°F, additional  $\gamma'$  must precipitate on cooling from this temperature or during subsequent aging at 1400°F. The room temperature hardness showed a slight increase by the addition of the second lower temperature age (fig. 48c). It was not determined from the photomicrographs whether this was due to a second finer dispersion of  $\gamma'$  or whether the majority of the  $\gamma'$  precipitated on the existing particles. Examination of tested specimens exhibited the following features which were similar for all three heat treated conditions:

- (1) The dislocation substructures (figs. 49 through 53) were similar to those previously presented for the material aged at 1700°F (figs. 43 through 47). The dislocations bowed between  $\gamma'$  particles, leaving pinched off dislocation loops (figs. 49 through 53). Gamma prime particles retained their coherency after creep-rupture testing (figs. 51a, 53c). Microtwins were also evident but to a lesser extent than was observed after aging at 1700°F. Again

it was evident that growth of the  $\gamma'$  occurred during the test exposures particularly at the higher temperatures and longer test times. For instance, during a 872 hour test at 1400°F and 38 ksi, the average size increased from about 600Å to 1200Å.

- (2) Considerable effort was spent in examination of specimens tested at 1200°F and 80,000 psi. The heat treatments caused a range of elongations at rupture. The material solution treated 4 hours at 1975°F and aged at 1550° and 1400°F, ruptured with 2.6 percent elongation whereas the material solution treated 1/2 hour at 1975°F and aged at 1550°F failed with 4.5 percent elongation. However, although the latter material exhibited a somewhat higher overall dislocation density, no other significant variations in dislocation structure were established (figs. 51, 52).

#### Materials Solution Treated At 1825°F

Heat treatment 1/2 hour at 1825°F did not result in complete solution of the  $\gamma'$  and, consequently, relatively large  $\gamma'$  particles (average diameter approximately 1200Å) were formed during this treatment. Subsequent aging at 1400°F produced a second, finer dispersion of  $\gamma'$ , thus resulting in a bimodal distribution of particle sizes. The finer  $\gamma'$  particles (established by transmission electron microscopy to be about 75Å in diameter) were not readily resolvable by replica techniques. The larger particles which developed during the 1825°F treatment were clearly visible (fig. 36a). For aging 10 hours at 1700°F



two individual size distributions were not discernible (fig. 37b). The average  $\gamma'$  size was somewhat larger than for the other materials aged at 1700°F.

For the material aged at 1400°F, the smaller particles grew during the test exposures at a greater rate than the larger particles. Consequently, with increasing time and temperature of the creep-rupture exposures the two  $\gamma'$  sizes merged tending towards a single distribution (fig. 54). Limited microstructural examination of tested specimens showed that the deformation was "general", i. e., the dislocations were homogeneously distributed (figs. 55, 56).

- (1) In the tests at the lower test temperatures there was evidence of stacking faults (fig. 55). Also, some superdislocations were observed. This would suggest the  $\gamma'$  particles were sheared by dislocations. However, the stacking faults were not present as highly extended ribbons, as was found for the material heat treated 1/2 hour at 1975°F and aged 16 hours at 1400°F. In addition, dislocations were observed bowing between larger particles and pinching off dislocation loops. These observations would suggest that the smaller particles of the bimodal size distribution are sheared by the dislocations while the large particles were by-passed. Presumably, the presence of the larger particles resulted in the homogeneous dislocation density since they forced the by-pass mechanism to be operative. Further structural examination would, however, be required to irrevocably establish the mechanisms for

this particular heat treated material.

- (2) From the study of specimens creep-rupture tested at 1200° and 1300°F (the higher test temperatures) no evidence was observed that indicated dislocations sheared  $\gamma'$  particles. The dislocation distributions were extremely homogeneous (fig. 56).

### Grain Boundary Structures

#### Grain Boundary Precipitates

All of the micrographs of the materials in the as-heat treated conditions showed the presence of grain boundary precipitates (figs. 35, 36). The amounts present tended to increase with the temperature of the solution treatment. For the materials solution treated at 1825°F and aged, the grain boundaries were partially filled with particles (figs. 36a, b). These were the only heat treated conditions which clearly showed evidence of precipitates in twin boundaries (figs. 35a, b). The grain boundaries were filled to an intermediate degree after the solution treatments at 1900° or 1975°F plus aging, with little difference between the two (figs. 36c, d, e, f). After solution treatment at 2150°F and aging, the boundaries were completely filled with precipitates (figs. 36g, h).

For the materials solution treated at 1975°F and particularly at 2150°F, the grain boundary precipitate consisted of two separate phases, carbides and  $\gamma'$  (figs. 57, 58). Both heat treatments apparently resulted in similar carbide distributions. The continuous nature of

the boundary phases for the material solution treated at the higher temperature was due to more extensive precipitation of  $\gamma'$ .

Although for each solution treatment, there may have been differences in the carbide morphology due to the aging treatments, none were characterized from the study carried out. Quantitative comparisons were difficult because the amount of carbide precipitate varied considerably from grain to grain. In addition, changes in the angle at which the plane of the sample intercepted the boundaries created differences in appearance.

#### Carbide Phase Identification By X-Ray Diffraction

X-ray diffraction patterns were obtained from bromine-extracted residues from each of the as-heat treated materials (table 13). These showed two compounds,  $\text{Ti(CN)}$  and  $\text{M}_{23}\text{C}_6$ , were present after each of the eight different heat treatments. The  $\text{Ti(CN)}$  phase is a cubic phase with a lattice parameter of  $4.32\text{\AA}$ , while  $\text{M}_{23}\text{C}_6$  has a face centered cubic structure and a lattice parameter of  $10.7\text{\AA}$ . No significant differences could be established between the relative intensities of corresponding lines with variations in heat treatment.

#### Orientation Relationships Of Grain Boundary Carbide

The orientation relationships and the identification of the phases present were established by transmission electron microscopy. From selected area diffraction patterns (fig. 59), the grain boundary carbide was shown to be  $\text{M}_{23}\text{C}_6$ . In addition, it was demonstrated that the carbide had the same orientation as one of the adjacent grains, i. e.,

$$\{100\}_{\gamma} \quad // \quad \{100\} M_{23}C_6$$

$$\langle 100 \rangle_{\gamma} \quad // \quad \langle 100 \rangle M_{23}C_6$$

This relationship arose since the carbide is face centered cubic with a lattice parameter ( $10.7\text{\AA}$ ) which is almost exactly three times that of the matrix ( $a_0 = 3.58\text{\AA}$ ). This situation is identical with that reported for the precipitation of  $M_{23}C_6$  in a  $\gamma'$  free austenite (ref. 10).

### Gamma Prime Depletion

Depletion of  $\gamma'$  in the areas adjacent to the grain boundaries occurred during heat treatment to a very limited extent for the material solution treated at  $2150^{\circ}\text{F}$  and aged 10 hours at  $1700^{\circ}\text{F}$  (fig. 57). The other heat treatments caused little or no  $\gamma'$  depletion. The occurrence of the depleted zones correlated with the occurrence of  $\gamma'$  in the grain boundaries. Presumably, the depleted regions resulted from the removal of the  $\gamma'$  forming elements from these areas and their precipitation in the grain boundaries.

Transmission electron micrographs of smooth specimens tested at  $1300^{\circ}$  and  $1400^{\circ}\text{F}$  showed that depletion of  $\gamma'$  had occurred during testing adjacent to the grain boundaries (fig. 60). The depletion was not detectably greater on boundaries transverse to the applied stress as was reported by Decker and Freeman (ref. 11). In many cases, the depleted areas occurred on only one side of the boundary (fig. 60). Electron diffraction patterns and dark field micrographs showed that the depleted side was that on which the  $M_{23}C_6$  carbide

did not have the identical orientation as the adjacent grain.

### Correlation Of The Time-Dependent Notch Sensitivity With The Dislocation Structure

The results suggest that a correlation exists between the time-dependent notch sensitivity and the predominant dislocation motion mechanism. Shearing of  $\gamma'$  by dislocations resulted in a greater susceptibility to notch sensitivity than when the  $\gamma'$  particles were bypassed. For materials aged at 1400°F after solution treatment at 1900°, 1975° or 2150°F, time-dependent notch sensitivity was observed only at the lower test temperatures. Little or no  $\gamma'$  growth occurred during these tests. The dislocations sheared the  $\gamma'$  particles. Growth of the  $\gamma'$  during the higher temperature creep-rupture tests resulted in a change of the dislocation motion mechanism to "by-passing". At the higher temperatures no time-dependent notch sensitivity was observed.

The materials aged 10 hours at 1700°F did not exhibit time-dependent notch sensitivity. The dislocations by-passed the  $\gamma'$  under all creep-rupture test conditions.

The material heat treated 1/2 hour at 1825°F plus 16 hours at 1400°F is a possible exception to the correlation. Time-dependent notch sensitivity was observed. The small  $\gamma'$  particles were sheared, however the larger particles were apparently by-passed by the dislocations.

In the following sections, mechanical characteristics that

controlled the time-dependent notch sensitivity are discussed with reference to the correlation.

### Yield Strengths:

Yield strengths which indicate the resistance of materials to dislocation motion in the early stage of tensile deformation can be considered in terms of the critical resolved shear stress necessary to initiate dislocation motion. Theory predicts that aging (for a constant volume fraction of  $\gamma'$ ) increases the critical resolved shear stress (CRSS) for dislocation motion until the particle dimensions exceed a critical size, after which further increase in size results in a decrease in the CRSS (ref. 12). Below the critical size the dislocations shear the particles while larger sizes are by-passed.

The size of the  $\gamma'$  particles relative to the critical for the as-heat treated materials were reflected in the yield strengths (at 1000°F, for example; table 3), room temperature hardnesses (fig. 48), and the structures observed by transmission electron microscopy (figs. 37 through 56):

- (1) For the material solution treated 1/2 hour at 1825°F and aged 16 hours at 1400°F, the  $\gamma'$  particles were both smaller and larger than the critical size. The combined effect, as reflected by the hardness tests, was an average size at about the critical, i. e., peak hardness. Aging 10 hours at 1700°F resulted in  $\gamma'$  larger than the critical and a lower CRSS.
- (2) For the materials solution treated at 1900°, 1975° and 2150°F

aging 16 hours at 1400°F resulted in  $\gamma'$  smaller than the critical with CRSS's higher than for aging 10 hours at 1700°F ( $\gamma'$  was larger than the critical). Aging 24 hours at 1550°F for the material solution treated at 1975°F resulted in  $\gamma'$  larger than the critical and a CRSS slightly below that obtained by aging 16 hours at 1400°F.

For each solution treatment, the time-dependent notch sensitivity that occurred after aging 16 hours at 1400°F, was not evident after aging to 10 hours at 1700°F. This increase in aging temperature resulted in lower yield strengths (or CRSS). The yield strength was also dependent on the solution treatment. Thus it was possible to lower the yield strength while utilizing the same aging treatment. For example, for aging at 1400°F solution treatment at 2150°F resulted in a much lower yield strength than for treatment at 1975°F (91 ksi and 115 ksi at 1000°F respectively). This decrease in yield strength was as large, if not larger, than obtained from the aging treatment variation. All materials aged at 1400°F exhibited time-dependent notch sensitivity. Consequently, lowering the yield strength by itself does not result in elimination of the notch sensitivity.

#### Deformation At Short Times

The experimental data demonstrated that aging at 1700° instead of 1400°F not only lowered the yield strength but it also increased the amount of creep deformation that occurred at short times. This was particularly evident at the lower test temperatures from the iso-creep strain curves previously presented (figs. 31 through 34). Many factors

can be expected to contribute to the creep characteristics at short times (ref 13). It must be assumed that a principal controlling factor is the nature of the interaction of the dislocations with the  $\gamma'$  particles. The results would indicate that shearing of the particles tends to limit the amount of deformation relative to when the by-pass mechanism is operative.

#### Accumulation of Damage

Failure by creep-rupture occurred by intergranular crack initiation and growth followed by transgranular fracture. The rupture times for the smooth specimens were primarily controlled by the time period for intergranular crack initiation. Consequently, metallurgical phenomena which contributed to intergranular crack initiation were "damaging" to the rupture properties. The transmission electron microstructural study of tested smooth specimens was carried out on samples taken somewhat removed from the fractures. The dislocation motion mechanisms observed were those which led to intergranular crack initiation. This was also true for the higher temperature tensile tests for which fracture was also initiated by intergranular cracking. The fractures for the lower temperature tensile tests were entirely transgranular. Consequently, the mechanisms observed were those which led to transgranular crack initiation.

The deformation that occurred in smooth specimens subsequent to crack initiation contributed relatively little to the overall elongation. This was because, after crack initiation, the deformation was highly



localized in the vicinity of the crack. Thus the elongation at rupture can be used as a measure of the creep damage necessary for crack initiation.

Deformation of the grains by localized slip causes incompatibilities at the grain boundaries which give rise to stress concentrations. Grain boundary sliding also results in high local stresses at the grain boundaries. If these processes continue, intergranular cracks will be initiated. Thus the inception of intergranular cracking will be dependent on (a) the nature of the intragranular deformation and (b) the metallurgical characteristics of the grain boundaries.

G. Luetiering and S. Weissman, in a study of the room temperature tensile properties of age-hardened Ti-Al and Ti-Cu alloys, demonstrated that a correlation existed between low ductility and particles being sheared by dislocations, whereas higher ductility resulted when the dislocations by-passed the precipitate particles (ref. 12). Analysis of the results for Waspaloy showed that similar correlations existed for both tensile and creep-rupture tests. For example, after solution treatment 1/2 hour at 1975°F aging 16 hours at 1400°F resulted in lower rupture elongations at the lower test temperatures than aging at 1700°F. These conditions correspond respectively to shearing and by-passing of the  $\gamma'$ . These observations are consistent with the concept that "homogeneous" deformation (resulting from the by-pass mechanism) leads to more ductile behavior since it avoids the high stress concentrations at the heads of

dislocation pile ups in the more localized deformation.

The heat treatment variations evaluated also resulted in differences in the grain boundary structures. These could also be expected to have influenced the observed variations in ductility:-

- (1) At the longer time tests at 1200°F the material heat treated 1/2 hour at 2150°F and aged 10 hours at 1700°F exhibited extremely low elongations at rupture (table 2; fig. 10). The average  $\gamma'$  size was considerably larger than the critical and consequently from the above considerations, high elongations would be expected. Therefore, for this heat treated material the initiation of inter-granular cracks at small amounts of creep deformation must be attributed to grain boundary characteristics. These clearly differed from those that occurred for the materials solution treated at lower temperatures. An extremely large grain size was evident (table 14). In addition, the grain boundaries were completely filled with precipitate, massive  $\gamma'$  and  $M_{23}C_6$  carbides.
- (2) The occurrence of a large grain size (table 14) for the material solution treated 4 hours at 1975°F and aged 24 hours at 1550°F plus 16 hours at 1400°F could also be correlated with the low elongations at rupture that were evident in the lower temperature rupture tests (table 10). Higher elongations (table 9; fig. 17) occurred for the material in the identical aged condition but solution treated only 1/2 hour at 1975°F. This latter material had a smaller grain size.

The influence of variations in grain boundary characteristics on the time-dependent notch sensitivity were not evident from the results. Presumably their effect was overshadowed by the very strong influence of the  $\gamma'$  precipitate. This apparently arose since the dislocation mechanism influenced not only the deformation characteristics but also the creep damage due to small creep strains.

## SUMMARY OF RESULTS

Several aspects of the scope and cause of time-dependent notch sensitivity were clarified by the present investigation of Waspaloy:

- (1) Time-dependent notch sensitivity of 0.026-inch thick sheet was observed to occur at temperatures from 900° to 1300°F. The results also indicated, however, that time-dependent notch sensitivity can be expected at prolonged times at lower temperatures. Notched to smooth rupture strength ratios as low as 0.44 were observed. This corresponds to the extremely low rupture time ratio of about  $1:10^6$ .
- (2) Variation in sheet thickness from 0.013 to 0.075-inch did not vary the severity of the time-dependent notch sensitivity for the heat treated condition evaluated (1/2 hour at 1975°F plus 16 hours at 1400°F).
- (3) Both smooth and notched creep-rupture specimens failed by relatively slow intergranular crack initiation and growth followed by transgranular fracture. The latter fracture occurred when the increase in stress on the load bearing area, due to the growth of the intergranular crack, exceeded that necessary to cause rapid shear. The occurrence of time-dependent notch sensitivity was due to premature initiation of intergranular cracks caused by localized creep deformation at the base of the notches. Critical to this crack initiation was yielding and subsequent creep that relaxed the high stresses introduced by the presence of the edge-notches.

- (4) Time-dependent notch sensitivity occurred when (i) the notched specimens were loaded below the approximate 0.2 percent smooth specimen offset yield strength; and (ii) test data from smooth specimens indicated that small amounts of creep strain used up large fractions of creep-rupture life. Most important, no reasons were evident why this result will not prove applicable not only to other gamma prime hardened materials but also for other alloy systems.
- (5) The occurrence of the notch sensitivity was dependent on the heat treatment. Of particular significance was the aging treatment, "overaging" resulted in elimination of the time-dependent notch sensitivity. For Waspaloy heat treated 1/2 hour at 1975°F plus 24 hours at 1550°F, the rupture strengths were high and no time-dependent notch sensitivity occurred in tests from 1000° to 1400°F.
- (6) Dislocations sheared particles smaller than a critical size. Particles larger than the critical were by-passed by the dislocations (except possibly at high strains). Shearing in contrast to by-passing was observed to result in smaller amounts of creep deformation at short times together with greater amounts of creep damage for small creep strains. These factors increased the susceptibility to notch sensitive behavior so that an apparent correlation existed between its occurrence and the nature of the dislocation motion mechanism operative.

## APPENDIX I

### TIME-TEMPERATURE RUPTURE CHARACTERISTICS

Time-dependent edge-notch sensitivity was observed at test temperatures of 900° to 1300°F, but to little or no extent in tests at 1400°F. It was also evident that, the lower the test temperature the longer the time period at which the time-dependent notch sensitivity occurred. This latter fact is consistent with the concept that effects of long time exposure at relatively low temperatures can often be simulated by a shorter time exposure at a higher temperature. The time-temperature relationships of the smooth and notched specimen rupture data were evaluated for the following reasons:-

- (1) To further characterize the variation of smooth and notched specimen creep-rupture behavior with time at temperatures from 1000° to 1400°F.
- (2) To predict the time periods at which the time-dependent notch sensitivity can be expected at test temperatures below 1000°F.

Several empirical methods have been used to correlate the rupture times of materials tested at a number of different temperatures (ref. 13). As a first step in examination of the time-temperature characteristics the most widely used technique, that of Larson-Miller (ref. 14) was applied to the rupture data. This method utilizes a parameter (P) which is plotted against the log of stress to establish a master curve for interpolation and extrapolation of data. A function of

time and temperature,  $P$  is defined by:

$$P = T(C + \text{Log } t)$$

where:  $T$  = Absolute temperature, °R

$t$  = time for rupture at  $T$ , hours

$C$  = Constant

The Larson-Miller parameter is widely used with a constant  $C$  of 20. This, however, is an "average" value so that a considerably better representation of any one data set can often be obtained by determining an optimum value of  $C$ . This has the effect of minimizing the standard deviation of the data from the master curve. In the present investigation optimization of  $C$  was accomplished by utilization of a computer program reported by Mendelson, Roberts and Manson (ref. 15).

Optimum constants were established for the heat treated conditions for which extensive stress-rupture time data were available for smooth and notched specimens (tables 5 through 8). Since time-temperature characteristics, as exemplified by parameter curves, can be compared for different materials only by use of a given fixed constant, the data were also evaluated utilizing a constant  $C$  of 20. In addition, the results of the "exploratory" smooth specimen tests carried out for a wide range of heat treated conditions (table 2) were also parameterized utilizing a value of 20 for  $C$ .

For the smooth specimen data, the optimum constants were fairly close to 20 and thus the parameter curves were similar to those

established using this latter "standard" value (table 15). Only the curves based on  $C=20$  have been included (figs. 61 through 66). From these the following factors were evident:

- (1) The rupture data consisted of two distinct regimes which differed significantly in slope. In general, the change in behavior from the high-stress to the low-stress regime occurred as the stress was reduced below the approximate 0.2 percent offset yield strength.
- (2) At the higher parameter values (i. e., higher temperatures and/or longer times) the rupture strengths were much less dependent on heat treatment than at the lower parameter values (except possibly the material solution treated at 2150°F and aged at 1400°F). The results of metallographic examination of the tested specimens indicated that the similarity of strengths resulted, at least in part, from gamma prime growth during the higher temperature tests.

Parameterization of notched specimen rupture data showed the two types of behavior discussed below. These were dependent on whether or not the material exhibited time-dependent notch sensitivity.

#### Materials Not Susceptible To Time-Dependent Notch Sensitivity

Optimum values of the Larson-Miller parameter constant were somewhat lower than those obtained for smooth specimen data (table 15). The mathematics of the parameter fitted the data reasonably well as was evident from the relatively low standard deviation values obtained. The rupture strengths for notched specimens were lower than for smooth at the lower parameter values (short test times and/or low



temperatures) (figs. 63,64). However, with increasing parameter values, the strengths of smooth and notched specimens became similar.

#### Materials Exhibiting Time-Dependent Notch Sensitivity

Optimum Larson-Miller constant values for the notched specimen data were significantly lower than obtained for the smooth specimen data (table 15). As the standard deviation values were high, it can be concluded that the mathematics of the parameter was not consistent with the data. This was also evident from the scatter of the parameterized data based on the optimum constants (fig. 67).

When parameterized utilizing a value of 20 for C no single curve could be drawn through the data. However, when the data from each temperature were considered, a distinctive pattern of behavior was evident (figs. 61,62). At the lower test temperatures, changes in slope occurred which were directly reflective of those previously described for the log stress-log rupture time characteristics. These changes in slope did not occur in the curves at the higher temperatures. Consequently, it can be concluded that the notched specimen behavior is not consistent with the parameter concept of trade off of temperature and time (hence the scatter in the parameterized data). Thus, when time-dependent notch sensitive behavior is evident at the lower temperatures, it does not necessarily occur at shorter times at higher temperatures. The results would indicate however, if a material exhibits time-dependent notch sensitivity at any given temperature, then it will also do so at longer times at lower temperatures.

Two questions arise concerning the prediction of notched specimen rupture times beyond the time periods of the data available.

- (1) Most important is the ability to predict the time periods at which time-dependent notch sensitivity might be expected at longer times and lower temperatures than those at which tests were conducted.
- (2) Determination of whether a decrease in notch sensitivity occurs at prolonged time periods at the lower test temperatures. Such a decrease was observed at the intermediate test temperatures.

Acceptable rupture strengths at prolonged times at the lower test temperatures were not obtained by application of the Larson-Miller parameter to entire data sets (for materials exhibiting time-dependent notch sensitivity). Research reported by Wilson et al (ref. 17) has shown that an alternative and somewhat superior method is by construction of iso-stress lines. This method entails the extrapolation of iso-stress lines as a family of curves on plots of log rupture time versus Temperature ( $T$ ) or reciprocal absolute temperature ( $1/T_{abs}$ ). The technique circumvents errors that may be introduced by the selection of particular parameter methods (i.e., of specific temperature-time relationships). It allows treatment of data that is apparently incompatible at low and high temperatures. Time-temperature data at constant stress levels taken from the notched specimen rupture curves for the materials solution treated 1/2 hour at 1825°F and 1/2 hour at 1975°F and aged 16 hours at 1400°F were

plotted on graphs of time versus  $T$  and  $1/T_{abs}$ . Typical examples are presented in Figures 68 and 69. Extrapolation of the curves for stress levels where time-dependent notch sensitivity occurred, provided estimates of rupture times at which these relatively low strengths could be expected at lower temperatures than those at which tests were conducted. For the material solution treated at  $1825^{\circ}\text{F}$ , it is evident that the 100,000-hour notched rupture strengths at  $700^{\circ}$  should be about 100,000 psi (figs. 68,69). Thus, extensive time-dependent notch sensitivity might be expected to occur in less than 100,000 hours at temperatures as low as  $700^{\circ}\text{F}$ . Similar analysis for the material solution treated at  $1975^{\circ}\text{F}$  indicated the occurrence of time-dependent notch sensitivity in less than 100,000 hours at temperatures as low as about  $850^{\circ}\text{F}$ .

Notched specimen rupture curves for the materials aged at  $1400^{\circ}\text{F}$ , exhibited a decrease in slope at intermediate test temperatures (figs. 12,13). The results of the microstructural study suggested that this occurred due to  $\gamma'$  growth during the test exposures. If this was indeed the case, then time-temperature trade-off concepts are applicable. They would indicate that similar breaks can be expected at prolonged time periods at lower temperatures. In addition, the iso-creep strain curves (figs. 31,32) would be expected to show a decrease in slope subsequent to the increase evident at the lower test temperatures.

## APPENDIX II

### INFLUENCE OF SHEET THICKNESS ON THE NOTCH SENSITIVITY

Tensile and creep-rupture tests from 1000° to 1400°F were carried out for smooth and notched specimens of 0.050-inch thick Waspaloy (W-II) sheet solution treated 1/2 hour at 1975°F and aged 16 hours at 1400°F (tables 16, 17). These tests were included in the program to provide an indication of the influence of sheet thickness on the mechanical characteristics. When compared with those for the 0.026-inch thick material (W-I) (table 6; fig. 13) they led to the following:-

- (1) Rupture strengths for smooth specimens from 1000° to 1400°F were slightly higher for the thicker material (fig. 70). Notched specimen rupture strengths for the 0.050-inch thick material were higher at 1000°, 1100° and 1200°F, and similar at 1400°F to those obtained from tests on the 0.026-inch thick material. At 1000°, 1100° and 1200°F the 0.026-inch thick material exhibited more severe time-dependent notch sensitivity than did the 0.050-inch thick material.
- (2) The results indicate that the differences in mechanical characteristics of the two materials would result in lower notch sensitivity for the thicker material. The 0.050-inch thick material had somewhat lower yield strengths (table 16) and consequently, the

stresses at notches must have been relaxed by "yielding" to lower levels than for the 0.026-inch thick material. Secondly, the life fraction for given small amounts of creep strain were (with the possible exception of 0.1 percent strain at 1200°F) lower for the thicker material (fig. 71). Relaxation of stress concentrations by creep would, therefore, result in less creep damage, and therefore, longer notched rupture lives for the thicker material.

Thus the observed variation in severity of time-dependent notch sensitivity with thickness increase from 0.026 to 0.050-inch was due, at least in part, to differences in the deformation characteristics of the two materials. These could have arisen from slight variation in composition, fabrication, or the materials reaction to the heat treatments. Obviously the observed effects also may have been influenced by the difference in sheet thickness.

As a first step in clarifying this situation, 0.050-inch thick material (W-II) was machined to 0.026-inches thick and heat treated 1/2 hour at 1975°F plus 16 hours at 1400°F prior to machining into notched specimens for rupture testing. The test results are presented as Table 18 and compared graphically with those obtained for the 0.050-inch thick specimens in Figure 72. Little or no variation in notched specimen strength occurred for sheet thickness difference of 0.026 to 0.050-inch.

In order to extend the scope of the above results, sheets 0.013,

0.020, 0.026, 0.038, 0.050, 0.062 and 0.075-inch thick were produced from 0.310-inch thick plate (W-III). The notch sensitive behavior of these materials, after heat treatment 1/2 hour at 1975°F plus 16 hours at 1400°F, was similar in nature to that previously described for the 0.026-inch thick material (W-I) commercially produced from the identical heat of Waspaloy. The following factors were evident from comparison of the creep-rupture data (table 19).

- (1) Smooth specimen rupture times were similar, independent of sheet thickness from 0.020 to 0.075 inches. The 0.013-inch thick material gave somewhat lower strengths which probably reflected the somewhat larger grain size for this material (table 14).
- (2) With consideration of the scatter evident in the notched specimen tests it was impossible to conclude that any major variation in rupture time occurred with sheet thickness variation. Rupture time is exceedingly sensitive to stress. Consequently, when the above differences with sheet thickness were compared in terms of the rupture strengths only very minor differences were evident. Therefore, within the range of sheet thickness from 0.013 to 0.075-inch little or no variation in severity of time-dependent notch sensitivity occurred.

## REFERENCES

1. Cullen, T.M. and Freeman, J.W.: "The Mechanical Properties at 800°, 1000° and 1200° of Two Superalloys Under Consideration For Use In The Supersonic Transport". Prepared under Grant No. NsG-124-61 (NASA CR-92) for NASA by The University of Michigan, Ann Arbor, September, 1964.
2. Cullen, T.M. and Freeman, J.W.: "The Mechanical Properties Of Inconel 718 Sheet Alloy at 800°, 1000° and 1200°F." Prepared under Grant No. NsG-124-61 (NASA CR-268) for NASA by The University of Michigan, Ann Arbor, July, 1965.
3. Raring, R.H.; Freeman, J.W.; Schultz, J.W.; and Voorhees, H.R.: "Progress Report of the NASA Special Committee On Materials Research For Supersonic Transports". NASA-TN-D-1798, May 1963.
4. Decker, R.F.; Rowe, J.P.; Bigelow, W.C.; Freeman, J.W.: "Influence Of Heat Treatment On Microstructure And High Temperature Properties Of A Nickel-Base Precipitation-Hardening Alloy". NACA TN 4329, July 1958.
5. Bigelow, W.C.; Amy, J.A.; Brockway, L.O.: "Electron Microscope Identification Of The Gamma Prime Phase Of Nickel-Base Alloys". Proc. ASTM, Vol. 56, p. 945, 1956.
6. Kelly, P.M.; Nutting, J.: "Techniques For The Direct Examination Of Metals By Transmission In The Electron Microscope". Journal Of Inst. of Metals, Vol. 87, 1958.
7. Dupres, A.T.: "A Study Of Dislocation Distributions In Annealed 306 Stainless Steel Produced By Fatigue Loading At Various Levels Of Strain And Numbers Of Cycles". Doctoral Thesis, University of Michigan, September 1963.
8. Wilson, F.G.; Pickering, F.B.: "Some Aspects Of The Deformation Of An Age-Hardened Austenitic Steel". Journal of Iron and Steel Institute, Vol. 207, Part 4, p. 490, 1969.
9. Kotval, P.S.: "The Microstructure Of Superalloys". Metallography Vol. I, p. 251, 1969.
10. Wolff, U.R.: "Orientation And Morphology Of  $M_{23}C_6$  Precipitated In High-Nickel Austenite". Trans. AIME, Vol. 236, p. 19, 1966.

11. Decker, R.F.; Freeman, J.W.: "The Mechanism Of Beneficial Effects Of Boron And Zirconium On Creep Properties Of A Complex Heat Resistant Alloy". Trans. AIME, Vol. 218, p. 277, 1960.
12. Luetjering, G.; Weissmann, S.: "Mechanical Properties Of Age-Hardened Titanium-Aluminum And Titanium-Copper Alloys". AFML-TR-69-15, 1969.
13. Decker, R.F.: "Strengthening Mechanisms In Nickel-Base Super-alloys." Presented at Steel Strengthening Mechanisms Symposium, Zurich, Switzerland, May 1969.
14. "Time-Temperature Parameter For Creep-Rupture Analysis". ASM Publication No. D8-100, October 1968.
15. Larson, F.R.; Miller, J.: "A Time-Temperature Relationship For Rupture And Creep Stresses". Trans. Amer. Soc. Mech. Engrs., Vol. 74, p. 754, 1952.
16. Mendelson, A.; Roberts, E.; Manson, S.S.: "Optimization Of Time-Temperature Parameters For Creep And Stress-Rupture, With Application To Data From German Cooperative Long-Time Creep Program". NASA-TN-D-2975, 1965.
17. Wilson, D.J.; Freeman, J.W.; Voorhees, H.R.: "Creep-Rupture Testing Of Aluminum Alloys To 100,000 Hours. Part I - Rupture Data For 1100-0 And 5454-0 Plate And A Modified Extrapolation Technique Used To Establish Stresses For The Initiation Of 15,000 And 100,000 Hour Tests". Prepared for the Metals Properties Council by The University of Michigan and Materials Technology Corporation, November 1969.



TABLE 1.

## TENSILE PROPERTIES OF 0.026-INCH THICK WASPALOY SHEET AT 1000°F

Smooth Specimen Properties								
Heat Treatment	Test Temp. (°F)	Tensile Strength (ksi)	0.2% Offset Yield Strength (ksi)	Y.S. T.S.	Elong. (%)	R. A. (%)	Notched Tensile Strength (ksi)	N/S Tensile Strength Ratio
1/2 hr. 1825°F								
+ 16 hrs 1400°F	1000	187.5	154.5	0.83	12.0	17	162.0	0.86
+ 16 hrs 1400°F	1000	187.2	155.8	0.83	11.3	20		0.86
+ 10 hrs 1700°F	1000	161.0	113.0	0.70	17.6	24	126.5	0.79
1/2 hr. 1900°F								
+ 16 hrs 1400°F	1000	163.8	113.0	0.69	28.9	31	134.7	0.82
+ 10 hrs 1700°F	1000	149.2	91.5	0.61	-	26	109.0	0.73
1/2 hr. 1975°F								
+ 16 hrs 1400°F	1000	161.0	115.0	0.71	30.0	32	132.5	0.82
+ 10 hrs 1700°F	1000	148.5	94.9	0.64	32.4	28	104.3	0.71
1/2 hr. 2150°F								
+ 16 hrs 1400°F	1000	134.0	91.0	0.68	26.5	26	112.0	0.84
+ 10 hrs 1700°F	1000	130.0	74.0	0.57	25.6	26	95.8	0.74

TABLE 2

SMOOTH SPECIMEN TENSILE AND CREEP RUPTURE PROPERTIES AT 1000° AND 1200°F FOR 0.026-INCH THICK WASPALOY SHEET (W-1)

	Test Temp	Stress	Rupture Time	Larson Miller Parameter	Elong.	R.A.	Min. Creep Rate	Inter. Crack Length		Test Temp	Stress	Rupture Time	Larson Miller Parameter	Elong.	R.A.	Min. Creep Rate	Inter. Crack Length
Heat Treatment	(°F)	(ksi)	(hrs.)	(C=20)	(%)	(%)	(%/hr.)	(%)	Heat Treatment	(°F)	(ksi)	(hrs.)	(C=20)	(%)	(%)	(%/hr.)	(%)
1/2 hr. 1825°F + 16 hrs. 1400°F	1000	187.5	Tens.		12.0	17		0	1/2 hr. 1975°F + 16 hrs. 1400°F	1200	164.3	Tens.		17.0	17		0
		187.2	Tens.		11.5	20		0			167.7	Tens.		19.5	20		0
		175	106.7	32.16	10.2	16	0.039	2			120	3.6	34.12	4.3	13		9
		165	479.8	33.11	4.4	12	0.0051	8			110	198.3	37.02	1.9	10	0.0012	17
		155	2540.6	34.17	2.5	8	0.00051	6			90	376.5	37.48	1.5	9	0.00045	23
	1200	185.7	Tens.		15.1	20		0	+ 10 hrs. 1700°F	1000	80	1240.8	38.38	2.2	10	0.000096	34
		120	57.7	36.12	5.1	7	0.064	5			80	1218.6	38.33	2.2	8	0.000142	37
		100	542.9	37.74	2.0	7	0.00162	42			140	82.7	32.10	23.8	22		0
		85	2560.3	38.86	3.4	8	0.00044	38			125	566.5	33.23	14.3	15	0.0027	7
											110	5469.0	34.67	8.9	11	0.000167	12
+ 10 hrs. 1700°F	1000	161.0	Tens.		17.6	24		0	1/2 hr. 2150°F + 16 hrs. 1400°F	1000	134.0	Tens.		26.5	26		0
		135	In progress 4867				-ve				120	93.0	32.08	14.9	19	0.0025	2
	1200	166.3	Tens.		17.7	17		0			110	370.5	32.96	6.2	16	0.000215	5
		95	322.9	37.35	5.5	10	0.0105	23			100	985.3	33.58	3.2	13	0.000017	7
	85	894.5	38.11	5.9	9	0.00372	28										
1/2 hr. 1900°F + 16 hrs. 1400°F	1000	163.8	Tens.		28.9	31		0	+ 10 hrs. 1700°F	1200	70	276.1	37.26	1.7	8	-ve	33
		150	113.0	32.20	11.0	16	0.00254	4			70	90.2 ph	36.46			<0.0003	-
		140	1060.8	33.66	6.8	15	0.00034	10			50	199.5 ph	37.02			<0.0003	-
	95	374.5	37.48	1.7	12	0.00035	23	50			1681.7	38.57	1.6	8	-ve	56	
+ 10 hrs. 1700°F	1000	140	86.9	32.03	20.8	20	0.0259	2	1/2 hr. 1975°F + 16 hrs. 1400°F	1000	161.0	Tens.		30.0	32		0
		130	350.5	32.93	14.5	14	0.00175	7			150	131.9	32.29	15.0	23	0.017	4
	1200	90	570.6	37.78	6.0	10	0.00181	23			145	207.6	32.58	13.5	20	0.00525	6
		75	2363.7	38.81	4.9		0.000237	43			135	517.0	33.17	7.0	14	0.00032	8

ph = failed at pin hole  
\* "parameter" tests

TABLE 3

NOTCHED SPECIMEN ( $K_t > 20$ ) TENSILE AND RUPTURE PROPERTIES AT 1000° AND 1200°F FOR 0.026-INCH THICK WASPALOY SHEET (W-1)

	Test Temp	Stress	Rupture Time	Intergranular Crack Length	N/S		Test Temp	Stress	Rupture Time	Intergranular Crack Length	N/S
Heat Treatment	(°F)	(ksi)	(hours)	(%)	Ratio	Heat Treatment	(°F)	(ksi)	(hours)	(%)	Ratio
1/2 hr. 1825°F						1/2 hr. 1975°F					
+ 16 hrs. 1400°F	1000	162.0	Tens.	0	.86	+ 16 hrs. 1400°F	1200	124.7	Tens.	0	.75
		135	227.7	13	.80			85	13.1	26	.73
		100	831.7	22	.62			70	42.5	29	.64
		80	884.1	25	.50			50	290.3	45	.47
		80	2913.6	40	.52			45	8643 Discontinued		>.71
	1200	145.0	Tens.	0	.78	+ 10 hrs. 1700°F	1000	104.3	Tens.	0	.71
		100	19.9	26	-			100	2296.0	9	.83
		80	253.2	36	.75			95	1203.0	10	.77
		65	4065.0	64	.78						
+ 10 hrs. 1700°F	1000	126.5	Tens.	0	.79		1200	104.2	Tens.	0	.72
		95	In progress 5347					85	164.9	30	.92
								70	928.1	33	.83
								45	4183.0 Discontinued		-
	1200	90	77.2	40	.83	1/2 hr. 2150°F					
		75	379.1	42	.81	+16 hrs. 1400°F					
		55	8459.5	54	.83		1000	112.0	Tens.	0	.87
								90	2572.5	19	.95
								75	5038.9	23	.83
1/2 hr. 1900°F							1200	65	59.5	46	.82
+ 16 hrs. 1400°F	1000	134.7	Tens.	0	.82			50	76.1 ph	-	>.64
		110	386.6	-	.70			50	179.3	47	.69
		85	2153.6	31	.62			45	164.3	50	.62
								35	4704.8 ph	-	>.85
	1200	75	82.2	38	.67	+ 10 hrs. 1700°F	1000	95.8	Tens.	0	.74
		65	174.2	44	.64			85	8112 Discontinued		>.86
		50	10,000 Discontinued		>.74			75	6145 Discontinued		>.75
+ 10 hrs. 1700°F	1000	109.0	Tens.	0	0.73		1200	85	42.6	13	.92
		95	7489 Discontinued		>.88			70	358.8 ph	-	.85
		85	6144.7 Discontinued		>.78			60	3917.9	77	.86
								60	4526.0		.87
1/2 hr. 1975°F						ph = failed at pin hole					
+ 16 hrs. 1400°F	1000	132.5	Tens.	0	.82						
		115	49.9	6	.82						
		110	728.0	12	.80						
		90	706.2	15	.65						
		85	2619.1	29	.66						
		70	4648.1	43	.56						

Table 4

TENSILE PROPERTIES OF 0.026-INCH THICK WASPALOY SHEET (W-1) AT  
1000°, 1200° AND 1400°F

Heat Treatment	Smooth Specimen Properties						Notched Tensile Strength (ksi)	N/S Tensile Strength Ratio
	Test Temp (°F)	Tensile Strength (ksi)	0.2% Offset Yield Strength (ksi)	$\frac{Y.S.}{T.S.}$	Elong. (%)	R. A. (%)		
1/2 hr. 1825°F + 16 hrs. 1400°F	1000	187.5	154.5	0.83	12.0	17		0.86
	1000	187.2	155.8	0.83	11.3	20	162.0	0.86
	1200	185.7	155.0	0.83	15.1	20	145.0	0.78
	1400	139.9	133.0	0.95	9.3	16	126.4	0.91
1/2 hr. 1975°F + 16 hrs. 1400°F	1000	161.0	115.0	0.71	30.0	32	132.5	0.82
	1200	164.3	114.5	0.70	17.0	17		0.76
	1200	167.7	111.3	0.67	19.5	20	124.7	0.74
	1400	125.0	102.0	0.82	5.8	16	117.5	0.94
1/2 hr. 1975°F + 10 hrs. 1700°F	1000	148.5	94.9	0.64	32.4	28	104.3	0.71
	1200	145.0	91.0	0.63	26.5	26	104.2	0.72
	1400	115.0	92.0	0.80	14.9	23	110.1	0.96
1/2 hr. 1975°F + 24 hrs. 1550°F	1000	166.3	111.5	0.67	25.6	28	121.5	0.73
	1200	162.2	111.5	0.69	22.8	30	118.4	0.73
	1400	116.8	-	-	14.3	21	121.2	1.04
1/2 hr. 1975°F + 24 hrs. 1550°F + 16 hrs. 1400°F	1000	170.9	117.5	0.69	23.4	25	114.3	0.67
	1200	164.0	109.0	0.67	22	28	121.1	0.74
	1400	109.5	108.0	0.99	13.8	24	119.8	1.09
4 hrs. 1975°F + 24 hrs. 1550°F + 16 hrs. 1400°F	1000	166.2	113.5	0.68	18.0	20	124.1	0.75
	1200	161.0	107.5	0.67	16.5	18	126.9	0.79
	1400	113.9	102.5	0.90	17.0	20	125.8	1.10

TABLE 5

SMOOTH AND NOTCHED(Kt=20) SPECIMEN TENSILE AND CREEP RUPTURE PROPERTIES AT 900°F to 1400°F FOR 0.026-INCH THICK WASPALOY SHEET (W-1)  
HEAT TREATED 1/2 HOUR AT 1825°F PLUS 16 HOURS AT 1400°F.

SMOOTH SPECIMENS								NOTCHED SPECIMENS					
Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter (C=20)	Elong. (%)	R. A. (%)	Min. Creep Rate (%/hr.)	Intergranular Crack Length (%)	Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter (C=20)	Intergranular Crack Length (%)	N/S Strength Ratio
950	180	489.3	31.99	14.1	18	0.0152	0.5	900	100	5530.3	32.29	20	
	175	754.0	32.26	8.2	17	0.0044	2						
1000	187.5	Tens.		12.0	17			950	155	Broke on loading.		0	
	187.2	Tens.		11.3	20		0		135	413.2	31.89	12	.71
	175	106.7	32.16	10.2	16	0.0390	2		110	1855.5	32.81	37	.65
	165	479.8	33.11	4.4	12	0.00510	8		100	2386.7	32.96	25	.59
	155	2540.6	34.17	2.5	8	0.00051	6	1000	162.0	Tens.		0	.86
1100	155	48.1	33.82	6.3	12		5		135	227.7	32.64	13	.80
	140	312.8	35.09	4.8	12	0.0107	16		100	831.7	33.46	22	.62
	120	3510.4	36.73	2.3	7	0.00029	23		80	884.1	33.52	25	.50
1200	185.7	Tens.		15.1	20		0		80	2913.6	34.26	40	.52
	120	57.7	36.12	5.1	7	0.064	5	1100	120	61.5	33.99	29	.78
	100	542.9	37.74	2.0	7	0.00162	42		100	156.0	34.62	27	.69
	85	2560.3	38.86	3.4	8	0.00044	38		80	379.1	35.22	40	.58
									65	1948.6	36.33	46	.52
1300	85	88.1	38.62	5.3	11	0.0294	27	1200	145.0	Tens.		0	.78
	60	1239.1	40.64	6.2	11	0.00214	58		100	19.9	35.36	26	-
	45.5	6708.3	41.93	7.2	15	0.00026	63		80	253.2	37.19	36	.75
1400	139.9	Tens.		9.3	16		8		65	4065.0	39.19	64	.78
	60	81.1	40.75	10.4	17		35	1300	65	233.3	39.37	51	.86
	40	722.2	42.52	7.5	14	0.0029	67		55	824.3	40.33	56	.7
								1400	126.4	Tens.		5	.91
									55	23.6	39.75	47	-
									40	475.5	42.18	60	.95

TABLE 6

SMOOTH AND NOTCHED ( $K_t \geq 20$ ) SPECIMEN TENSILE AND CREEP RUPTURE PROPERTIES AT 1000° TO 1400°F FOR 0.026-INCH THICK WASPALOY SHEET (W-1)  
HEAT TREATED 1/2 HOUR AT 1975°F PLUS 16 HOURS AT 1400°F.

SMOOTH SPECIMENS								NOTCHED SPECIMENS					
Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter (C=20)	Elong. (%)	R. A. (%)	Min. Creep Rate (%/hr.)	Intergranular Crack Length (%)	Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter (C=20)	Intergranular Crack Length (%)	N/S Strength Ratio
1000	161.0	Tens.		30.0	32		0	1000	132.5	Tens.		0	.82
	150	131.9	32.29	15.0	23	0.017	4		115	49.9	31.68	6	.75
	145	207.6	32.58	13.5	20	0.00525	6		110	728.0	33.38	12	.80
	135	517.0	33.17	7.0	14	0.00032	8		90	706.2	33.3	15	.65
	130	1953.5	34.02	4.6	11	0.00004	10		85	2619.1	34.19	29	.66
	130	2837.6	34.25	4.5	12	0.000045	11		70	4648.1	34.55	43	.56
1100	135	83.1	34.20	4.7	12	0.0025	10	1100	85	168.8	34.67	27	.65
	120	1461.2	36.14	2.5	12	0.00016	14		70	332.1	35.13	28	.55
	120	1047.6	35.94	2.8	12	0.0003	19		50	3153.4	36.66	42	.44
	59.5	3700	Discontinued			-ve							
1200	164.3	Tens.		17.0	17		0	1200	124.7	Tens.		0	.75
	167.7	Tens.		19.5	20		0		85	13.1	35.05	26	.73
	120	3.6	34.12	4.3	13		9		70	42.5	35.90	29	.64
	110	198.3	37.02	1.9	10	0.0012	17		50	290.3	37.29	45	.47
	90	376.5	37.48	1.5	9	0.00045	23		45	8643.0	Discontinued	-	>.71
	80	1240.8	38.38	2.2	10	0.000096	34	1300	55	16.6	37.35	40	.58
	80	1218.6	38.33	2.2	8	0.000142	37		50	947.6	40.44	46	.88
									45	2216.7	41.09	52	.88
1300	80	63.7	38.37	1.7	7	0.005	44	1400	117.5	Tens.			.94
	65	302.4	39.57	1.8	7	0.00060	50		95	0.1	35.34	10	-
	55	1288.5	40.67	5.1	10	0.00038	60		50	71.0	40.64	67	.81
	35	11,000	Discontinued			0.000043			45	260.7 ph	41.69	-	.96
1400	125.0	Tens.		5.8	16		8		38	1066.8	42.83	73	1.00
	65	18.1	39.53	2.8	9		38		38	1136.4	42.88	74	1.01
	45	406.1	42.04	6.5	11	0.0025	55						
	38	1006.9	42.78	8.1	15	0.00053	-						

TABLE 7

SMOOTH AND NOTCHED ( $K_t > 20$ ) SPECIMEN TENSILE AND CREEP RUPTURE PROPERTIES AT 1000° TO 1400°F FOR 0.026-INCH THICK WASPALOY SHEET (W-1)  
HEAT TREATED 1/2 HOUR AT 1975°F PLUS 10 HOURS AT 1700°F.

SMOOTH SPECIMENS								NOTCHED SPECIMENS					
Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter (C=20)	Elong. (%)	R.A. (%)	Min. Creep Rate (%/hr.)	Intergranular Crack Length (%)	Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter (C=20)	Intergranular Crack Length (%)	N/S Strength Ratio
1000	148.5	Tens.		32.4	28		0	1000	104.3	Tens.		0	.71
	140	82.7	32.10	23.8	22		1		100	2296.0	34.11	9	.83
	125	566.5	33.23	14.3	15	0.0027	7		95	1203.0	33.70	10	.77
	110	5469.0	34.67	8.9	11	0.000167	12						
1100	125	36.4	33.64	17.5	17	0.0650	4	1100	90	23.6	33.34	9	.71
	100	366.2	35.21	6.8	12	0.0028	16		85	1753.6	36.26	28	.89
	100	1184.7	36.00	6.9	11	0.00138	20	1200	104.2	Tens.		0	.72
	59.5	3740	Discontinued	-	-	-ve	-		85	164.9	36.88	30	.92
1200	145.0	Tens.		26.5	26		0		70	928.1	38.13	33	.83
	100	38.7	35.83	7.5	12	0.0692	17		45	4183	Discontinued	-	-
	80	1870.0	38.63	5.4	11	0.00018	32	1300	70	114.3	38.82	48	.96
1300	100	7.9	36.77	8.4	12		11		55	895.5	40.40	55	.95
	80	52.6	38.23	5.6	11		28	1400	110.1	Tens.		10	.96
	60	671.6	40.18	7.6	10	0.00112	40		55	43.2	40.24	56	.93
	38.5	5150	Discontinued			0.000088			38	791.3	42.59	61	.99
1400	115.0	Tens.		14.9	23		12						
	60	35.0	40.17	12.0	15		44						
	38	931.1	42.72	9.0	14	0.0011	52						
	26.6	3838.2	43.87	13.0		0.00015	-						

TABLE 8

SMOOTH AND NOTCHED ( $K_t \geq 20$ ) SPECIMEN TENSILE AND CREEP RUPTURE PROPERTIES AT 1000° TO 1400°F FOR 0.026-INCH THICK WASPALOY SHEET (W-1)  
HEAT TREATED 1/2 HOUR AT 1975°F PLUS 24 HOURS AT 1550°F.

SMOOTH SPECIMENS								NOTCHED SPECIMENS					
Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter (C=20)	Elong. (%)	R.A. (%)	Min. Creep Rate (%/hr.)	Intergranular Crack Length (%)	Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter (C=20)	Intergranular Crack Length (%)	N/S Strength Ratio
1000	166.3	Tens.		25.6	28		0	1000	121.5	Tens.		0	.73
	155	52.2	31.71	24.0	21		0.5		105	1797.8	33.95	16	.83
	135	615.7	33.27	11.7	13	0.00287	15	1100	105	131.6	34.52	13	.82
1100	120	425.9	35.30	5.2	8	0.00583	12		90	645.9	35.59	23	.77
1200	162.2	Tens.		22.8	30		1	1200	118.4	Tens.		0	.73
	95	580.0	37.79	5.2	8	0.00133	36		90	135.2 ph	36.73	-	.86
	80	2483.0	38.84	4.5	8	0.00012	40		80	972.4	38.17	43	.91
1300	80	134.2	38.94	7.2	12	0.016	30		70	3508.9	39.09	50	.92
	55	2090.8	41.04	3.8	9	0.000460	59	1300	80	46.6	38.13	49	.87
1400	116.8	Tens.		14.3	21		12		60	762.1	40.28	54	.95
	45	423.5	42.09	7.4	14	0.00373	52	1400	121.2	Tens.		10	1.04
	38	872.5	42.67	9.3	15	0.00102	56		38	1146.5	42.88	76	1.03



TABLE 9

SMOOTH AND NOTCHED ( $K_t > 20$ ) SPECIMEN TENSILE AND CREEP RUPTURE PROPERTIES AT 1000° TO 1400°F FOR 0.026-INCH THICK WASPALOY SHEET (W-1)  
HEAT TREATED 1/2 HOUR AT 1975°F PLUS 24 HOURS AT 1550°F PLUS 16 HOURS AT 1400°F.

SMOOTH SPECIMENS								NOTCHED SPECIMENS					
Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter (C=20)	Elong. (%)	R. A. (%)	Min. Creep Rate (%/hr.)	Intergranular Crack Length (%)	Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter (C=20)	Intergranular Crack Length (%)	N/S Strength Ratio
1000	170.9	Tens.		23.4	25		0	1000	114.3	Tens.		0	.67
	155	97.7	32.11	15.9	18	0.0664	1		110	3136.0	34.32	20	.91
	135	624.2	33.29	7.0	8	0.00373	7						
1100	120	333.8	35.15	6.0	8	0.0096	14	1100	105	252.0	34.96	25	.88
	106.1	3440.6	36.73		9	0.00040	22		90	6261.8	37.13	39	.88
1200	164.0	Tens.		22.0	28		0	1200	121.1	Tens.		0	.74
	95	281.8	37.27	6.1	10	0.0138	34		80	627.4	37.85	35	.90
	80	2679.7	38.87	5.0	10	0.00027	32		70	2424.8	38.82	46	.89
1300	80	100.3	38.73	6.2	9		37	1300	80	43.4 ph	38.08	-	>.90
									60	825.0 ph	40.33	-	>.97
1400	109.5	Tens.		13.8	24		-	1400	119.8	Tens.		10	1.09
	45	547.2	42.28	13.0	17	0.0023	56		38	862.7	42.67	76	1.08
	38	1195.6	42.92	12.6	15	0.00094	66						

TABLE 10

SMOOTH AND NOTCHED ( $K_t > 20$ ) SPECIMEN TENSILE AND CREEP RUPTURE PROPERTIES AT 1000° TO 1400°F FOR 0.026-INCH THICK WASPALOY SHEET (W-1)  
HEAT TREATED 4 HOURS AT 1975°F PLUS 24 HOURS AT 1550° PLUS 16 HOURS AT 1400°F.

SMOOTH SPECIMENS								NOTCHED SPECIMENS					
Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter (C=20)	Elong. (%)	R.A. (%)	Min. Creep Rate (%/hr.)	Intergranular Crack Length (%)	Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter (C=20)	Intergranular Crack Length (%)	N/S Strength Ratio
1000	166.2	Tens.		18.0	20		0	1000	124.1	Tens.		0	.75
	155	75.7	31.94	15.1	16	0.0917	0.5		110	930.1	33.53	16	.85
	135	495.0	33.14	8.2	11	0.0054	-		105	5131.7	34.63	21	.91
	130	859.3	33.48	7.4	11	0.00295	7		90	2238.4	34.10	23	.74
	120	In progress	2900			0.000215							
1100	120	211.9	34.83	7.6	12	0.0206	13	1100	105	461.0	35.37	19	.89
	110	1753.1	36.27	4.9	9	0.00177	24		90	2702.0	36.73	32	.87
	105	2980.1	36.63	3.8	8	0.00040	20	1200		Tens.			.79
1200		Tens.							80	394.0	37.52	30	.86
	95	240.6	37.17	2.0	8	0.00385	21		70	4255.5	39.23	40	.95
	80	1706.0	38.62	2.6	9	-	35		60	6748.1	39.56	61	.85
	74.5	3494.2	39.08	2.1	7	0.00005	41	1300	80	39.0	38.00	40	.90
1300	80	89.2	38.63	6.3	12	0.0275	38		60	474.6	39.92	57	.90
	55	2436.1	41.16	5.3	7	0.00031	46	1400	125.8	Tens.		6	1.10
1400	113.9	Tens.		17.0	20		7		45	350.2	41.93	65	.98
	45	380.1	41.99	6.9	12	0.00104	54		38	1026.6	42.80	71	1.05
	38	944.4	42.73	7.8	13	0.0005	52						

TABLE 11

SMOOTH AND NOTCHED ( $K_t > 20$ ) SPECIMEN CREEP-RUPTURE PROPERTIES AT 1000° TO 1400°F FOR 0.026-INCH THICK WASPALOY SHEET (W-IV) ANNEALED AND AGED 16 HOURS AT 1400°F

SMOOTH SPECIMENS								NOTCHED SPECIMENS					
Test Temp (°F)	Stress (ksi)	Rupture Time (hours)	Larson-Miller Parameter (C = 20)	Elong. (%)	R. A. (%)	Min. Creep Rate (% / hr.)	Intergranular Crack Length (%)	Test Temp (°F)	Stress (ksi)	Rupture Time (hours)	Larson-Miller Parameter (C = 20)	Intergranular Crack Length (%)	N/S Stress Ratio
1000	145 L	157.3	32.41	10.4	16	0.0067	2	1000	115 L	Broke on loading		0	-
	135 L	420.3	33.03	6.3	11	0.000665	6	100 L	895.1	33.51		23	0.70
	135 L	577.4	33.24	5.7	11	0.000585	7	80 L	1039.1	33.61		23	.60
	125 L	2592.4	34.19	3.3		0.0000065	8	60 L	2637.0	34.20		36	.47
	105 L	1005. Discontinued		-	-	-ve -	-	115 T	301.3	32.82		12	.81
	150 T	227.4	32.64	7.4	10	0.00222	6	100 T	383.6	32.98		20	.71
	145 T	181.3	32.50	10.0	-	0.00438	6	80	1797 Discontinued				>.62
	135 T	2650.0	34.18	3.3	7	0.00002	7	60	2128.2	34.06		39	.46
	125 T	2916.8	34.27	3.2	12	0.000048	9						
								1200	80 L	22.4	35.44	22	.65
								60 L	149.4	36.81		-	.59
								55 L	19.5	35.39		21	.45
1200	140 L	1.4	33.45	6.6	15	-	5	50 L	980.3	38.16		48	.63
	120 L	14.5	35.13	2.8	10	0.062	7	80 T	21.4	35.42		19	.65
	100 L	127 ph	36.69	-	-	-	-	60 T	117.7	36.63		35	.58
	100 L	40.1	35.86	1.1	7	0.00176	21	55 T	28.6	35.62		31	.45
	100 L	40.1 ph	35.86	-	-	-	-	50 T	883.9	38.10		47	.62
	70 L	1674.0	38.55	1.0		0.000072	33						
	140 T	1.4	33.45	5.5	9	-	-	1400	50 L	1.9	37.72		-
	120 T	19.5	35.34	2.0	7	-	10	45 L	3.3	38.16		51	-
1400	110 T	35.3	35.77	0.8	7	-	14	40 L	4.0	38.32		-	-
								15 L	3912 Discontinued				
								38 T	4.2	38.36		46	
								35 T	256.0	41.67		77	.81
								30 T	1028.2	42.80		80	.82
	50 L	71.2	40.63	2.8	8	0.0099	55						
	38 L	744.0	42.54	7.1	11	0.00106	81						

ph = Failed at pin hole

L = Longitudinal specimen

T = Transverse specimen

TABLE 12

SMOOTH AND NOTCHED SPECIMEN CREEP-RUPTURE PROPERTIES AT 1000° TO 1400°F FOR 0.026-INCH THICK WASPALOY SHEET (W-IV) ANNEALED AND AGED 16 HOURS AT 1400°F.  
SMOOTH SPECIMENS PRE-STRAINED SMALL AMOUNTS AND NOTCH SPECIMENS LOADED TO 115 ksi PRIOR TO CREEP-RUPTURE TESTING (LONGITUDINAL SPECIMENS).

SMOOTH SPECIMENS									NOTCHED SPECIMENS					
Plastic Pre-Strain (%)	Test Temp. (°F)	Stress (ksi)	Rupture Time (hours)	Larson- Miller Parameter (C=20)	Elong. (%)	R. A. (%)	Min. Creep Rate (%/hr.)	Intergranular Crack Length (%)	Test Temp. (°F)	Stress (ksi)	Rupture Time (hours)	Larson- Miller Parameter (C=20)	Intergranular Crack Length (%)	N/S Strength Ratio*
**	1000	135	500.5	33.16	12.2	16	0.00116	3	1000	95	817.3	33.46	17	0.71
0.25	1200	90	165.4	36.88	1.3	7	0.00055	21		88	1132.0	33.67	18	.67
0.49		90	153.1	36.82	1.8	8	0.00085	15		80	9721.8	Discontinued	-	>.67
2.2		70	1172.3	38.28	3.8	8	0.00365	29		75	7704.0	Discontinued	-	>.62
0.45	1400	45	41.6	40.20	1.9	6	0.012	50	1200	70	360.1	37.45	43	.77
1.60		38	146.9	41.23	--	--	0.00452	50		60	2186.5	38.74	36	.97
0.61		35	352.8	41.93	2.5	7	0.00195	-		50	6759.6	39.56	46	.98
									1400	45	70.0	40.63	48	.92
										38	153.2 ph.	41.27	-	>.86

ph = failed at pin hole

\* = Ratio of notch strength to smooth specimen strength (for no pre-loading).

\*\* = Pre-loaded to 145,000 psi

TABLE 13

X-RAY DIFFRACTION DATA OF EXTRACTED RESIDUES  
OF WASPALOY IN VARIOUS HEAT TREATED CONDITIONS

Solution Treatment	½ hr at 1825°F	½ hr at 1825°F	½ hr at 1900°F	½ hr at 1900°F	½ hr at 1975°F	½ hr at 1975°F	½ hr at 2150°F	½ hr at 2150°F	Standard Patterns for Indicated Phases			
Aging Treatment	16 hrs at 1400°F	10 hrs at 1700°F	16 hrs at 1400°F	10 hrs at 1700°F	16 hrs at 1400°F	10 hrs at 1700°F	16 hrs at 1400°F	10 hrs at 1700°F				
									TiC a <sub>0</sub> = 4.32		M <sub>23</sub> C <sub>6</sub> a <sub>0</sub> = 10.7	
Intensity	"d"	"d"	"d"	"d"	"d"	"d"	"d"	"d"	"d"	Intensity	"d"	Intensity
s	2.49	2.49	2.49	2.50	2.50	2.49	2.49	2.49	2.49	m		
s	2.38	2.39	2.39	2.40	2.39	2.39	2.39	2.39			2.39	s
s*		2.18		2.19		2.18		2.18			2.18	s
s*	2.16	2.16	2.15	2.16	2.16	2.14	2.16		2.16	vs		
vs	2.054	2.058	2.051	2.065	2.056	2.058	2.054	2.052			2.055	s
w	1.888	1.891	1.888	1.900	1.888	1.889	1.887	1.886			1.888	m
w	1.803	1.805	1.802	1.813	1.806	1.81	1.808	1.806			1.805	s
vvw		1.786				1.781	1.784				1.78	mw
vw	1.605	1.614		1.616	1.611	1.629	1.611				1.61	vw
w	1.524	1.529	1.525	1.533	1.529	1.518	1.527		1.525	s		
vvw		1.338			1.337	1.339		1.336			1.335	w
s	1.298	1.301	1.31	1.303	1.305	1.301	1.304	1.297	1.305	m	1.30	s
s	1.260	1.264	1.261	1.264	1.261	1.261	1.253	1.259			1.258	ms
m		1.237	1.248	1.240	1.249		1.248		1.25	m		

\* Two lines which were difficult to separate in the patterns.

TABLE 14

## GRAIN SIZES OF WASPALOY SHEETS IN THE AS-HEAT TREATED CONDITIONS

Material	Thickness (inch)	Condition	Average Grain Diameter (MM)
W-I	0.026	Solution treated 1/2 hour at 1825°F and aged	0.019
		Solution treated 1/2 hour at 1900°F and aged	0.019
		Solution treated 1/2 hour at 1975°F and aged	0.022
		Solution treated 1/2 hour at 2150°F and aged	0.13
		Solution treated 4 hours at 1975°F and aged	0.10
W-II	0.050	Solution treated 1/2 hour at 1975°F and aged	0.026
W-III	0.013	Hot rolled 2000°F, solution treated 1/2 hour	0.051
	0.020	at 1975°F and aged	0.021
	0.026	" " "	0.020
	0.038	" " "	0.022
	0.050	" " "	0.026
	0.062	" " "	0.024
	0.075	" " "	0.022

TABLE 15

SUMMARY OF OPTIMIZED STRESS - RUPTURE PARAMETER CONSTANTS FOR 0.026-INCH THICK WASPALOY SHEET (W-1) IN HEAT TREATED CONDITIONS AS DETERMINED BY APPLICATION OF THE LARSON-MILLER PARAMETER

<u>Heat Treatment</u>	<u>SMOOTH SPECIMENS</u>				<u>NOTCHED SPECIMENS</u>			
	<u>No. of Data Points</u>	<u>Larson- Miller Constant C</u>	<u>Standard Deviation</u>	<u>Degree of Polynomial</u>	<u>No. of Data Points</u>	<u>Larson- Miller Constant C</u>	<u>Standard Deviation</u>	<u>Degree of Polynomial</u>
<u>1/2 hr. 1825°F</u> + 16 hrs. 1400°F	16	22.9	0.088	6	18	8.2	0.336	1
<u>1/2 hr. 1975°F</u> + 16 hrs. 1400°F	19	19.1	0.344	3	19	11.5	0.415	7
+ 24 hrs. 1550°F	9	18.5	0.055	6	9	14.0	0.205	4
+ 10 hrs. 1700°F	14	17.0	0.221	2	10	15.8	0.189	6

TABLE 16

TENSILE PROPERTIES AT 1000°, 1200° AND 1400°F OF WASPALOY SHEETS 0.050 (W-11) AND 0.026 (W-1)  
INCHES THICK HEAT TREATED 1/2 HOUR AT 1975°F AND AGED 16 HOURS AT 1400°F.

Smooth Specimen Properties								
Sheet Thickness (inches)	Test Temp (°F)	Tensile Strength (ksi)	0.2% Offset Yield Strength (ksi)	<u>Y.S.</u> <u>T.S.</u>	Elong. (%)	R. A. (%)	Notched Tensile Strength (ksi)	N/S Tensile Strength Ratio
0.026	1000	161.0	115.0	0.71	30.0	32	132.5	0.82
	1200	164.3	114.5	0.70	17.0	17	124.7	0.76
	1200	167.7	111.3	0.67	19.5	20		0.74
	1400	125.0	102.3	0.82	5.8	16	117.5	0.94
0.050	1000	158.5	109.0	0.69	34.2	-	128.7	0.81
	1200	162.2	108.5	0.67	24.7	27	126.9	0.78
	1400	120.9	100.0	0.83	10.0	17	113.5	0.94



TABLE 17

SMOOTH AND NOTCHED (K1>20) SPECIMEN TENSILE AND CREEP RUPTURE PROPERTIES AT 1000° TO 1400°F FOR 0.050-INCH THICK WASPALOY SHEET (W-II)  
HEAT TREATED 1/2 HOUR AT 1975°F PLUS 16 HOURS AT 1400°F

SMOOTH SPECIMENS								NOTCHED SPECIMENS					
Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter (C=20)	Elong. (%)	R. A. (%)	Min. Creep Rate (%/hr.)	Intergranular Crack Length (%)	Test Temp (°F)	Stress (ksi)	Rupture Time (hrs.)	Larson-Miller Parameter C=20	Intergranular Crack Length (%)	N/S Strength Ratio
1000	158.5	Tens.		34.2			0	1000	128.7	Tens.		0	0.81
	150	145.1	34.37	20.9		0.0067	3		111	896.5	33.52	14	0.80
	145	323.5	32.87	16.2	22	0.0044	6		105	1717.0	33.93	21	0.77
	140	475.5	33.37	14.7	17	0.0011	7		95	2017.2	34.05	28	0.70
	130	In progress	9107			0.00033			85	2964.0	34.27	34	0.64
1100	120	420.8	35.32	4.9	11	0.0006	13	1100	95	209.0	34.82	21	0.77
1200							0.5		82.2	411.7	35.29	39	0.69
	162.2	Tens.		24.7	27			70	537.8	35.47	36	0.59	
	90	513.1	37.71	2.6	11	0.000183	26	65	1672.8	36.23	44	0.58	
	80	1692.1	38.57	3.4	10	0.00007	43						
1300								1200	126.9	Tens.			0.78
	63	569.0	40.06	10.2	21	0.00107	45	85	32.6	35.72			
	55	2615.0	41.22	21.6	25	0.00034	39	70	57.5	36.12	27	0.67	
								65	160.8	36.87	50	0.66	
1400	120.9	Tens.		10.0	17		7		58.8	9960	Discontinued		>0.84
	65	23.3	39.73	9.6	15		36						
	38	1715.3	43.22	16.2	23	0.00035	38	1400	113.5	Tens.		15	0.94
								65	2.9	38.05	32		
								45	467.2	42.16	64	1.02	

TABLE 18

NOTCHED SPECIMEN ( $K_t > 20$ ) RUPTURE PROPERTIES AT 1000° TO 1200° FOR 0.026-INCH THICK WASPALOY SHEET MECHANICALLY THINNED FROM 0.050-INCH THICK MATERIAL (W-II) AND SUBSEQUENTLY HEAT TREATED 1/2 HOUR AT 1975°F PLUS 16 HOURS AT 1400°F.

<u>Test Temp (°F)</u>	<u>Stress (ksi)</u>	<u>Rupture Time (hrs.)</u>	<u>Larson Miller Parameter (C=20)</u>	<u>Inter. Crack Length (%)</u>	<u>N/S Strength Ratio</u>
1000	85	4817.4	34.58	26	0.65
1100	85	524.1	33.46	31	0.71
	70	647.6	33.59	41	0.59
	60	1121.3	35.97	44	0.52
1200	70	118.6	36.63	28	0.63
	55	1241.6	38.58	41	0.67

TABLE 19

SMOOTH AND NOTCHED ( $K_t > 20$ ) SPECIMEN CREEP RUPTURE PROPERTIES AT 1000° TO 1400°F FOR 0.013 TO 0.075-INCH THICK  
WASPALLOY SHEETS (W-III) HEAT TREATED 1/2 HOUR AT 1975°F PLUS 16 HOURS AT 1400°F

SMOOTH SPECIMENS								NOTCHED SPECIMENS				
Sheet Thickness (inches)	Test Temp (° F)	Stress (ksi)	Rupture Time (hours)	Elong. (%)	Q. A. (%)	Min. Creep Rate (%/ hr. )	Intergranular Crack Length (%)	Sheet Thickness (inches)	Test Temp (° F)	Stress (ksi)	Rupture Time (hours)	Intergranular Crack Length (%)
0.013	1000	140	58.3	9.9	17	-	4	0.026	1000	94.7	574.6	15
0.020			4174.8	5.5	11	0.000145	12					
0.026			1307.0	5.7	14	0.00128	12	0.013			872.0	30
0.050			1427.6	11.6	16	0.00105	8	0.020			1233.1	21
0.062			817.5	12.2	17	0.00115	6	0.038			1316.4	20
								0.062			2381.3	28
0.013	1100	115	147.3	1.9	10	0.00055	15	0.013	1100	85	327.2	31
0.038			3308.2	2.1	8	0.000066	17	0.020			249.1	23
0.075			2812.7	2.2	9	0.000060	14	0.026			456.3	28
0.013	1200	90	19.5ph	-	-	-	-	0.026	1200	70	186.8	27
0.013			88.0ph	-	-	-	-	0.050			215.6	29
0.020			721.8	3.2	9	0.00030	28	0.075			226.6	33
0.026			861.1	4.3	11	0.00032	23					
0.050			261.0ph	-	-	(0.00037)	-	0.013			567.0	28
0.075			1038.7	3.2	9	0.00025	22	0.013			596.6	39
											0.020	1249.0
0.013	1400	45	10.1sh	0.5	-	-	-	0.026	1400	56.6	234.6	24
0.020			150.7	3.7	12	0.0059	75	0.038			748.5	37
0.038			411.7	12.3	20	0.0022	45	0.062			590.9	32
0.062			501.6	13.7	23	0.00195	36					
								0.026	1100	56.6	3414.3	35
ph = failed at pin hole								0.013	1200	70	35.4	39
sh = failed at shoulder								0.020			2082.7	34
								0.020			15.5	26
								0.026			88.1	39
								0.038			124.7	35
								0.050			65.2	35
								0.075			107.7	36
								0.013	1200	50	111.7	41
								0.020			1072.1ph	-
								0.026			In progress 1200	
								0.038			1630.3 Discontinued	
								0.075			1401.7	47

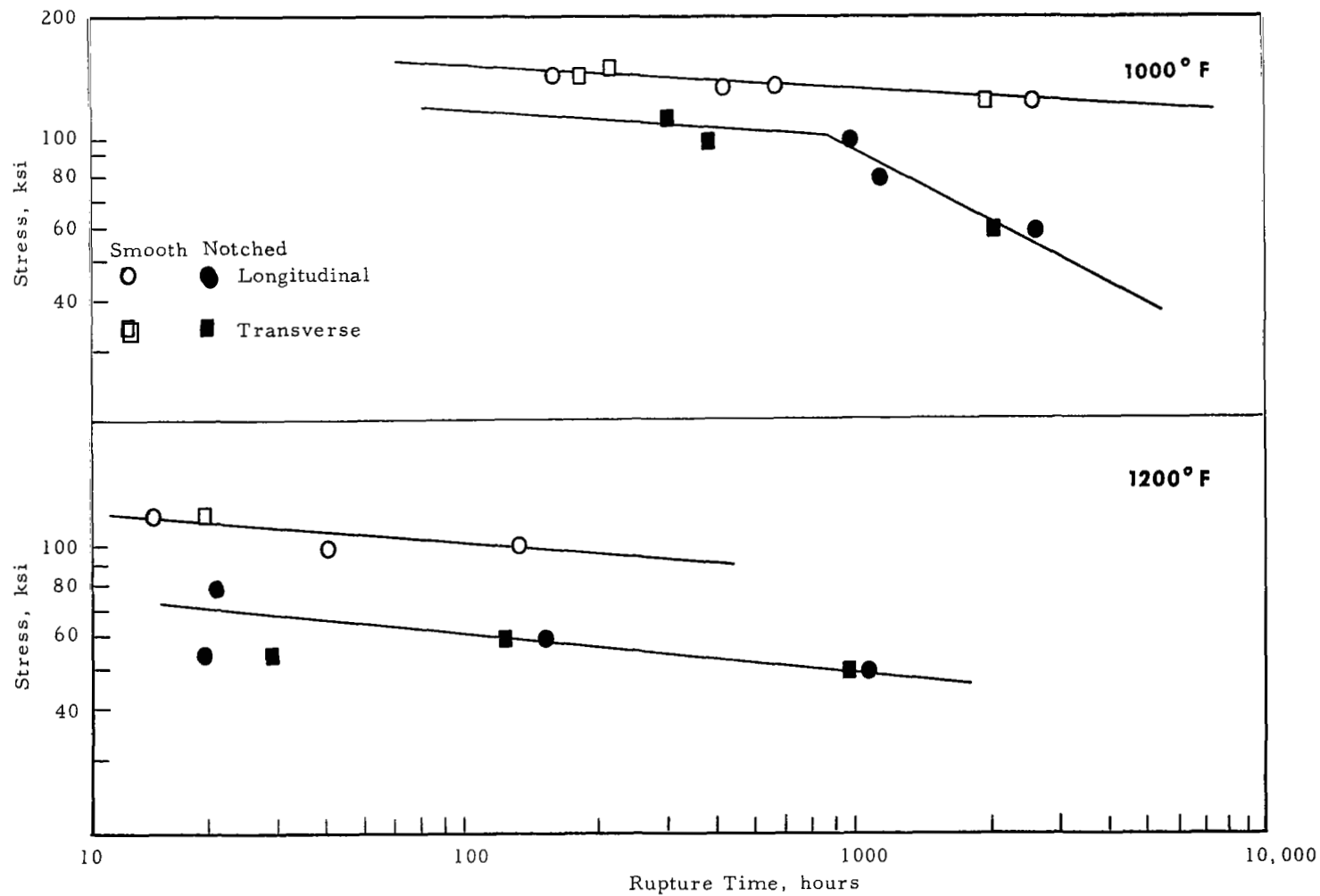


Figure 1. Stress versus rupture time data at 1000° and 1200°F obtained from smooth and edge-notched specimens of 0.026-inch thick Waspaloy sheet in the annealed and aged condition (ref. 1). A time-dependent increase in notch sensitivity is evident at 1000°F. Low notched to smooth rupture strength ratios occur at prolonged times at 1000°F and in the tests at 1200°F.

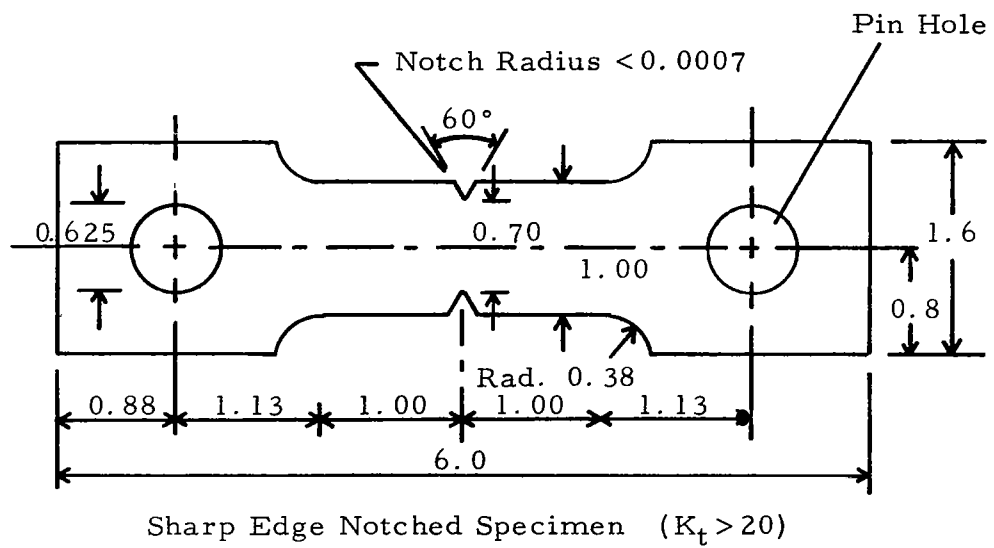
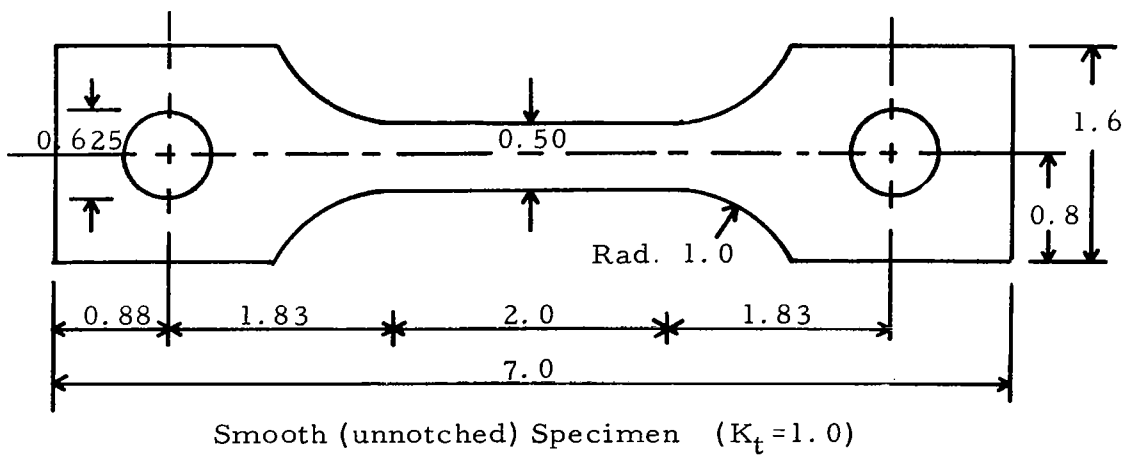


Figure 2. Types of test specimens (all dimensions in inches).

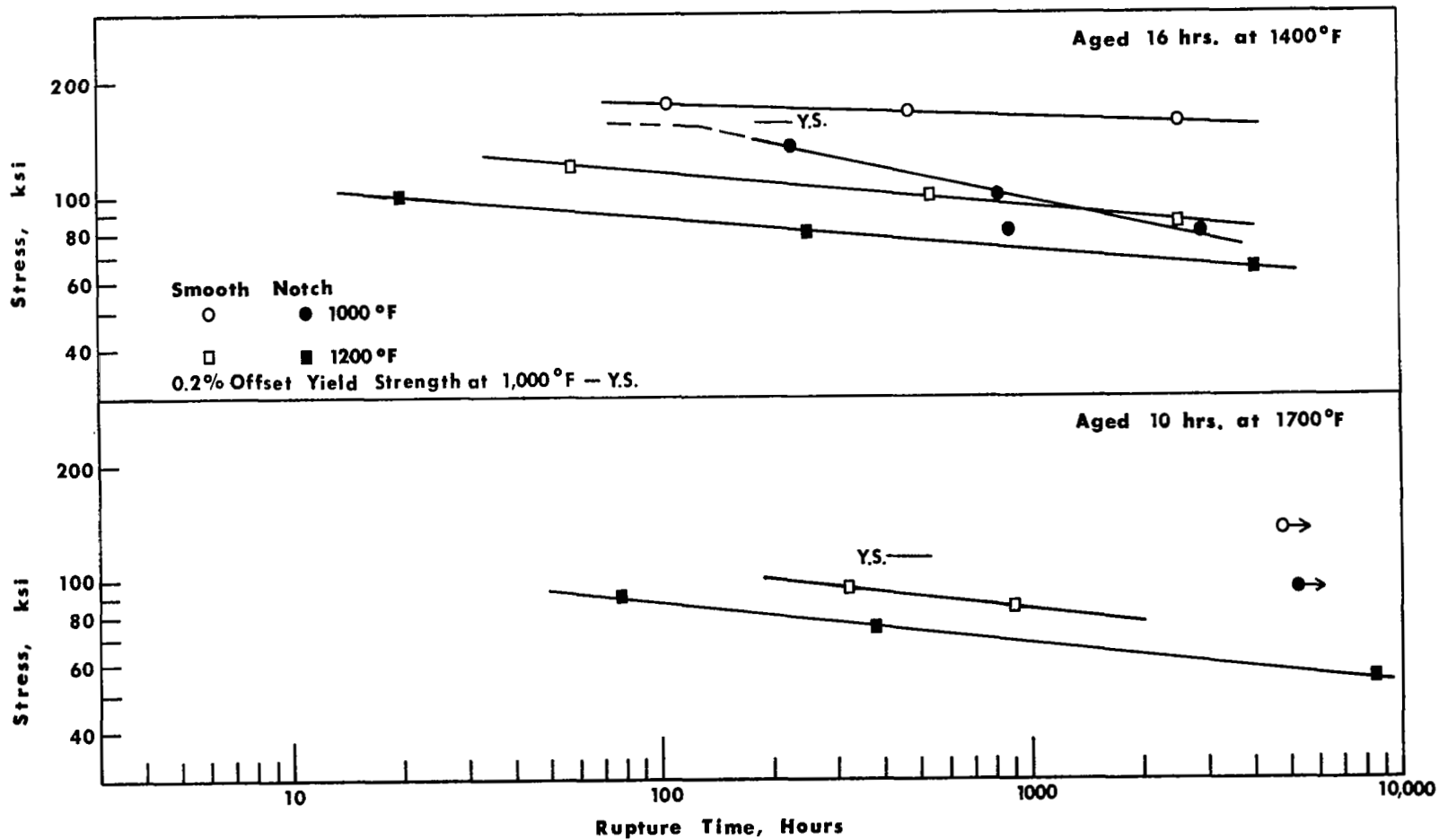


Figure 3. Stress versus rupture time data at 1000° and 1200°F obtained from smooth and notched specimens of 0.026-inch thick Waspaloy sheet solution treated 1/2 hour at 1825°F and aged. For aging at 1400°F time-dependent notch sensitivity is evident at 1000°F but not at 1200°F. The tests show no time-dependent notch sensitivity after aging at 1700°F.

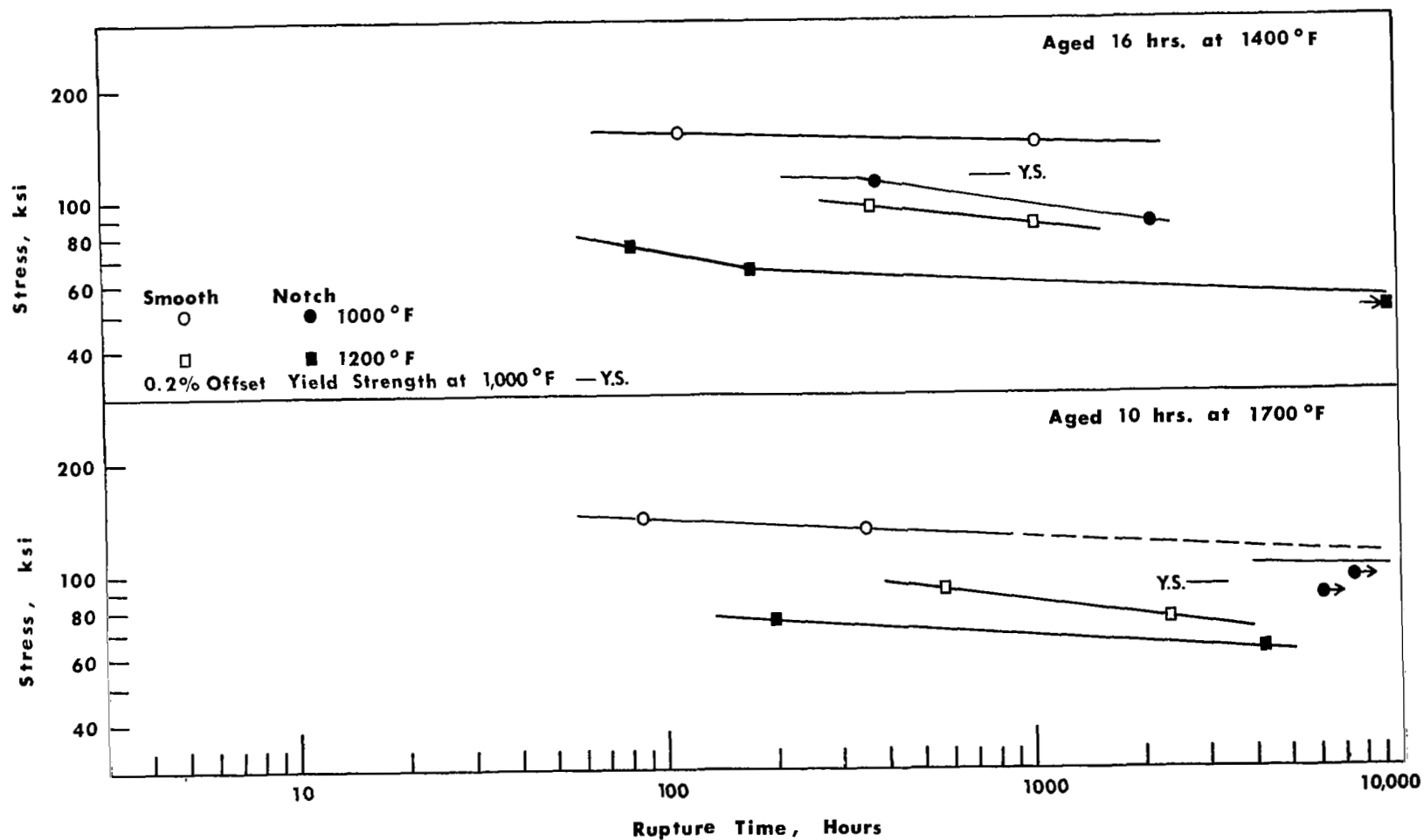


Figure 4. Stress versus rupture time data at 1000° and 1200°F obtained from smooth and notched specimens of 0.026-inch thick Waspaloy sheet solution treated 1/2 hour at 1900°F and aged. For aging at 1400°F the notch sensitivity increases at prolonged times at 1000°F, increases and subsequently decreases with time at 1200°F. No time-dependent notch sensitivity is evident after aging at 1700°F.

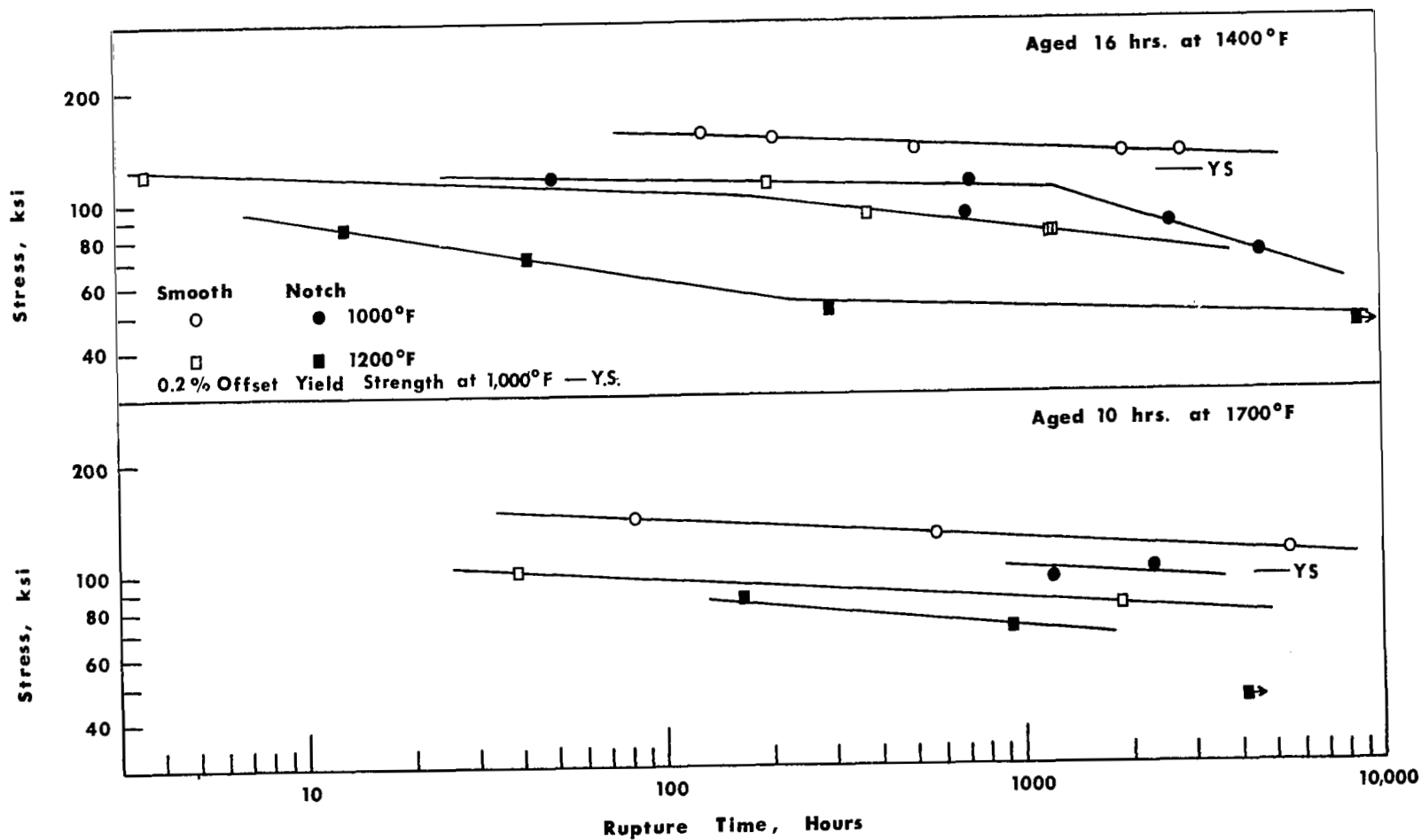


Figure 5. Stress versus rupture time data at 1000° and 1200°F obtained from smooth and notched specimens of 0.026-inch thick Waspaloy sheet solution treated 1/2 hour at 1975°F and aged. Time-dependent notch sensitivity is evident for aging at 1400°F but not for 1700°F in a similar manner as shown for solution treatment at 1900°F (see Figure 4).



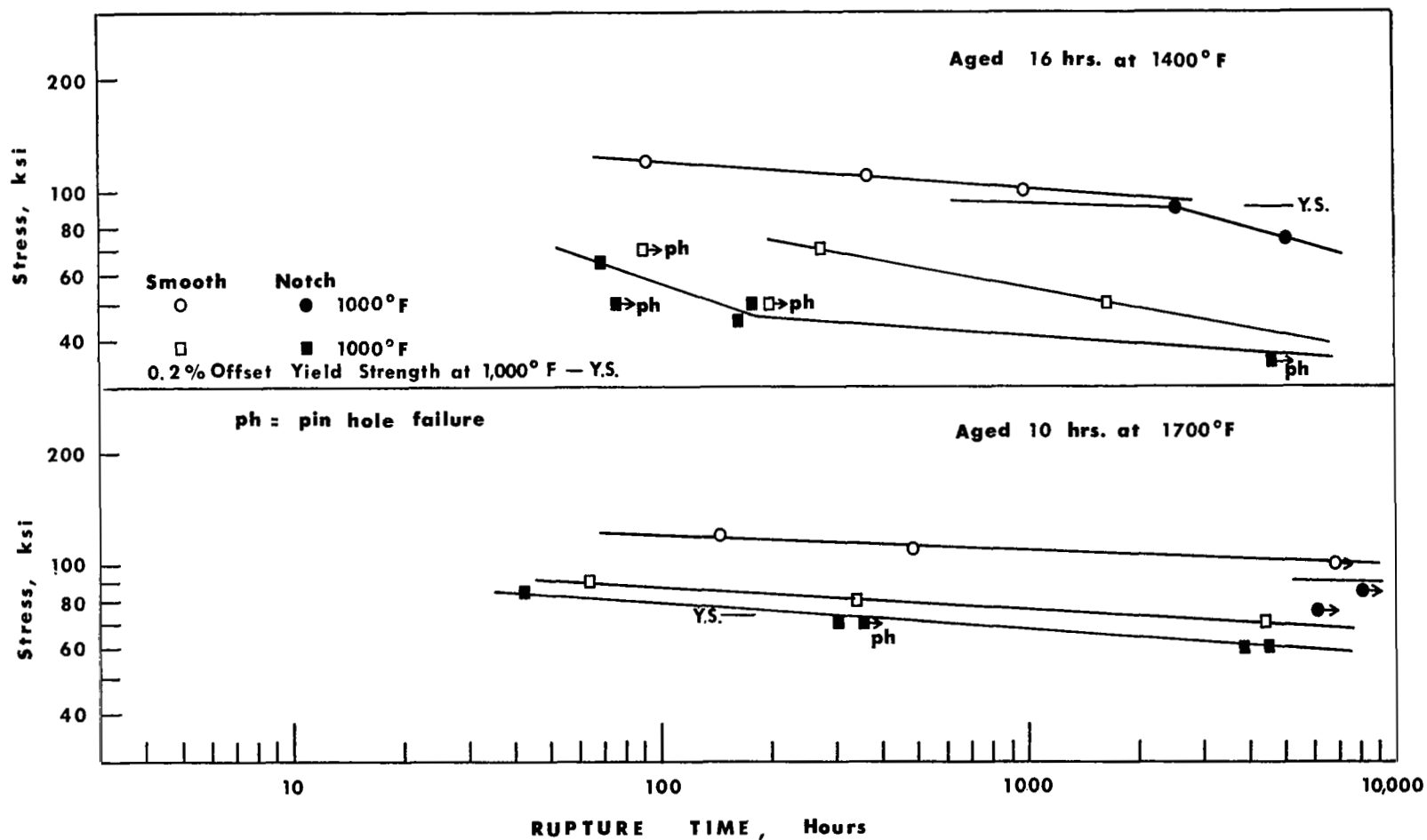


Figure 6. Stress versus rupture time data at 1000° and 1200°F obtained from smooth and notched specimens of 0.026-inch thick Waspaloy sheet solution treated 1/2 hour at 2150°F and aged. Time-dependent notch sensitive behavior is similar to that for solution treatments at 1900° and 1975°F.

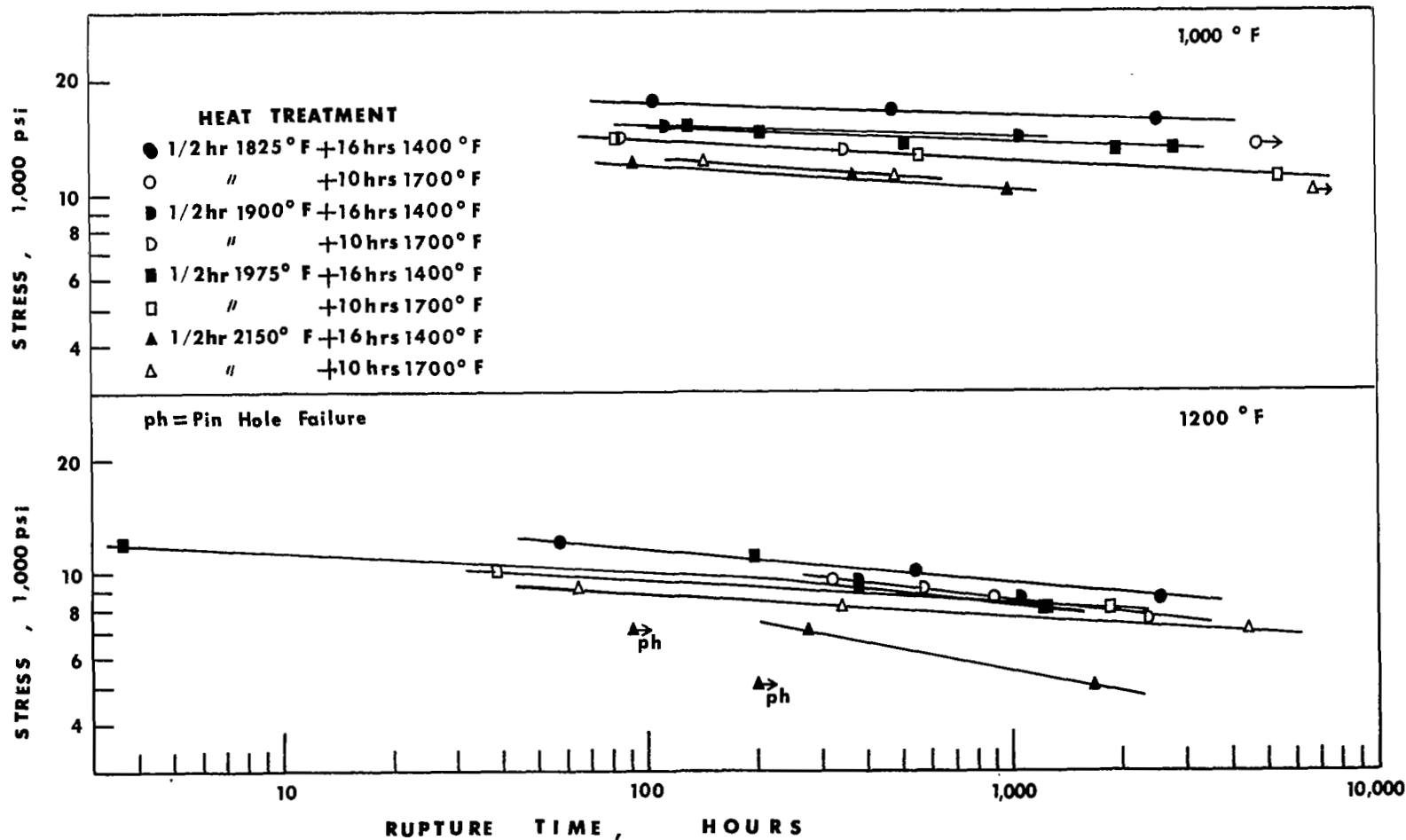


Figure 7. Stress versus rupture time data at 1000° and 1200°F obtained from smooth specimens of 0.026-inch thick Waspaloy in the heat treated conditions.

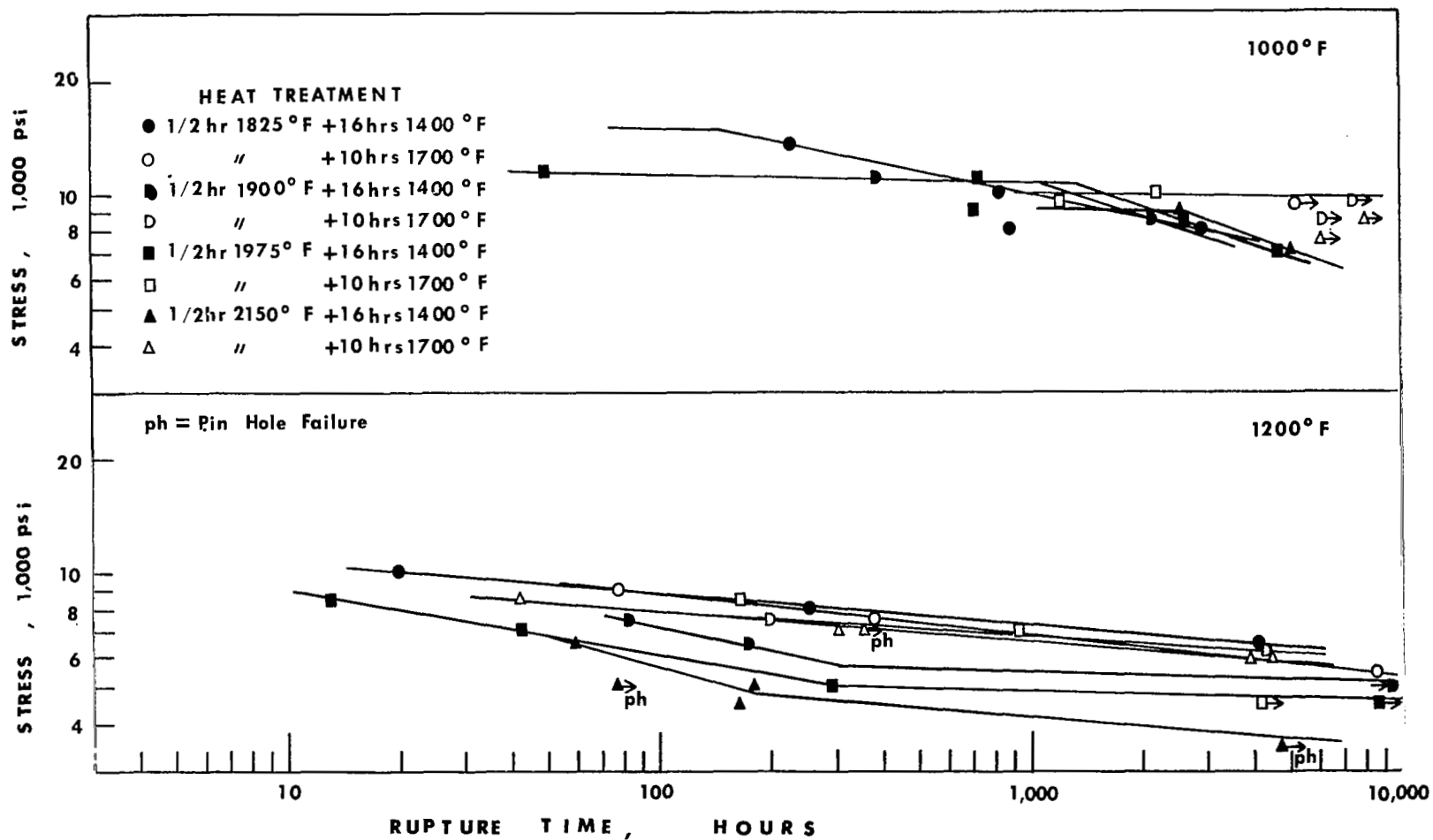


Figure 8. Stress versus rupture time data at 1000° and 1200°F obtained from notched specimens of 0.026-inch thick Waspaloy in the heat treated conditions.

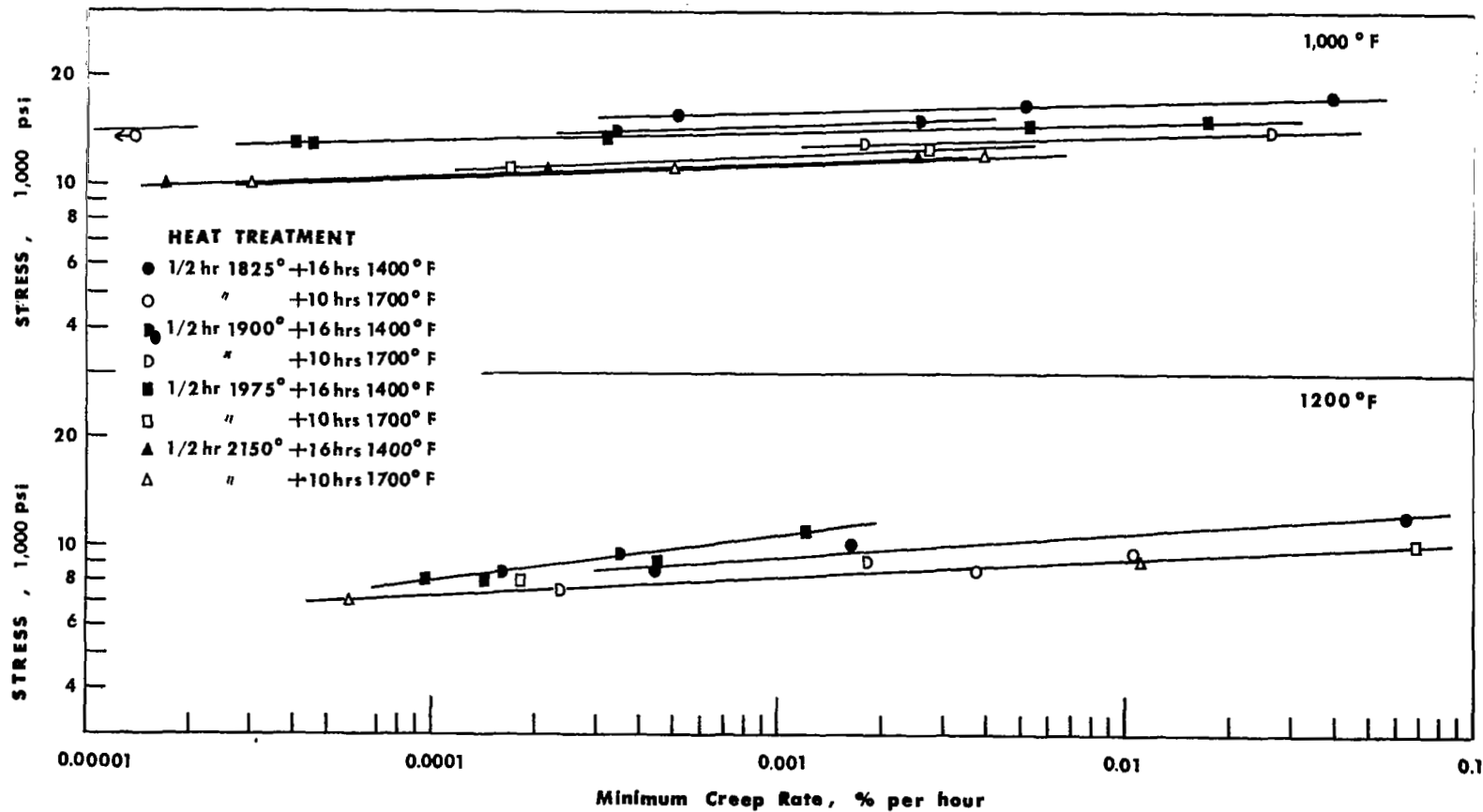


Figure 9. Stress versus minimum creep rate behavior at 1000° and 1200°F for 0.026-inch thick Waspaloy sheet in the heat treated condition.

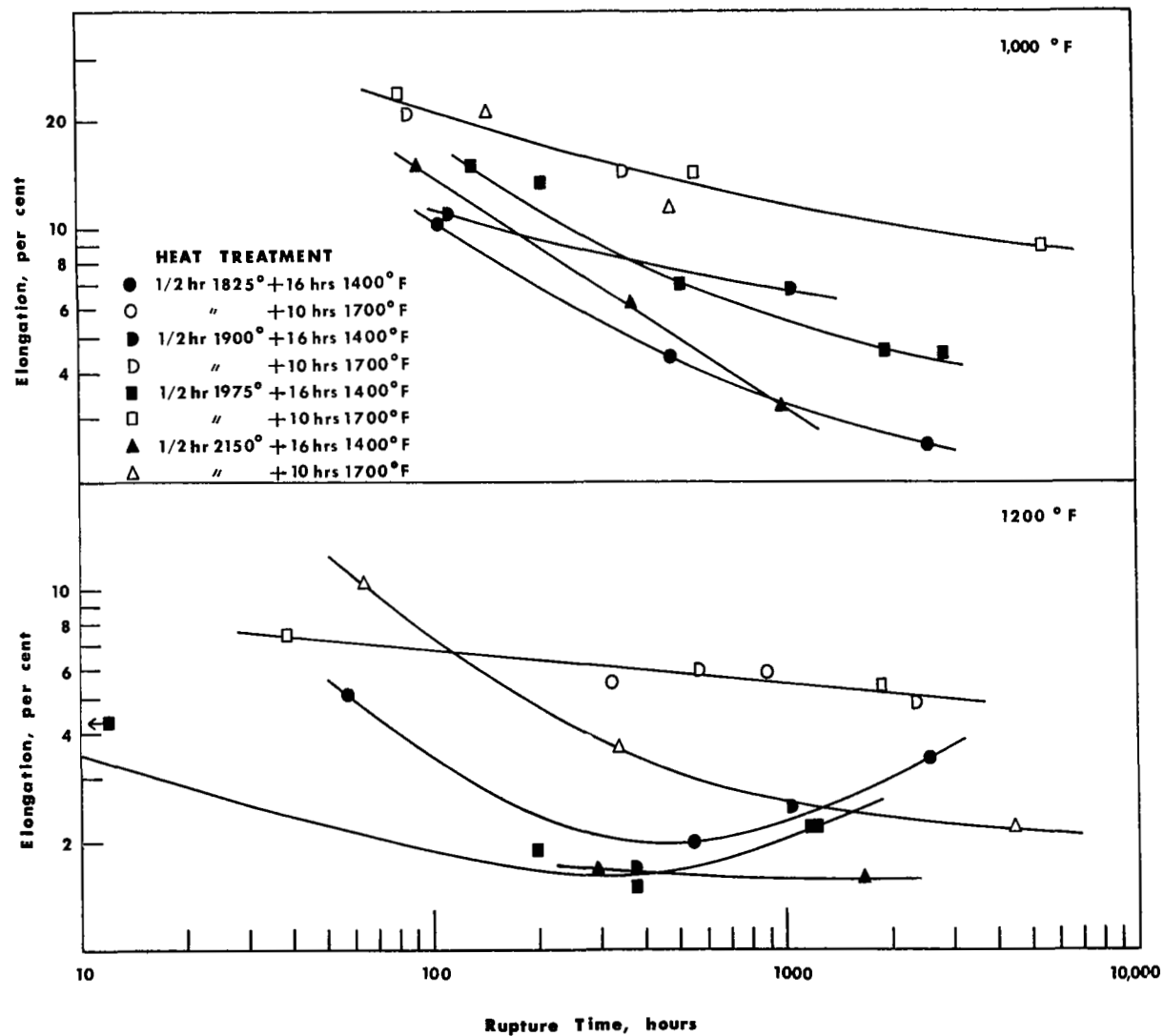


Figure 10. Elongation versus rupture time data at 1000° and 1200°F for smooth specimens of 0.026-inch thick Waspaloy sheet in the heat treated condition.

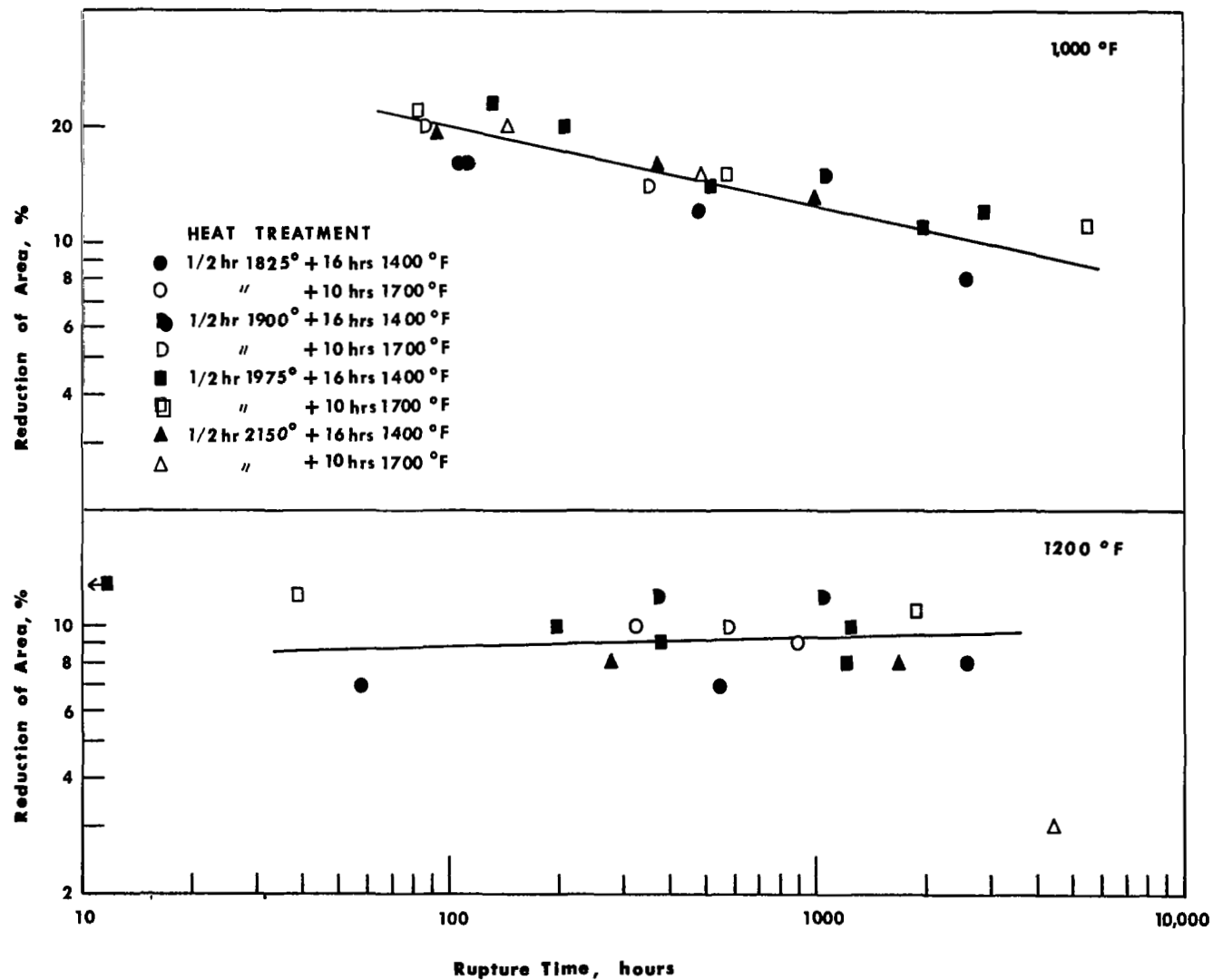


Figure 11. Reduction in area versus rupture time data at 1000° and 1200°F for smooth specimens of 0.026-inch thick Waspaloy sheet in the heat treated condition.

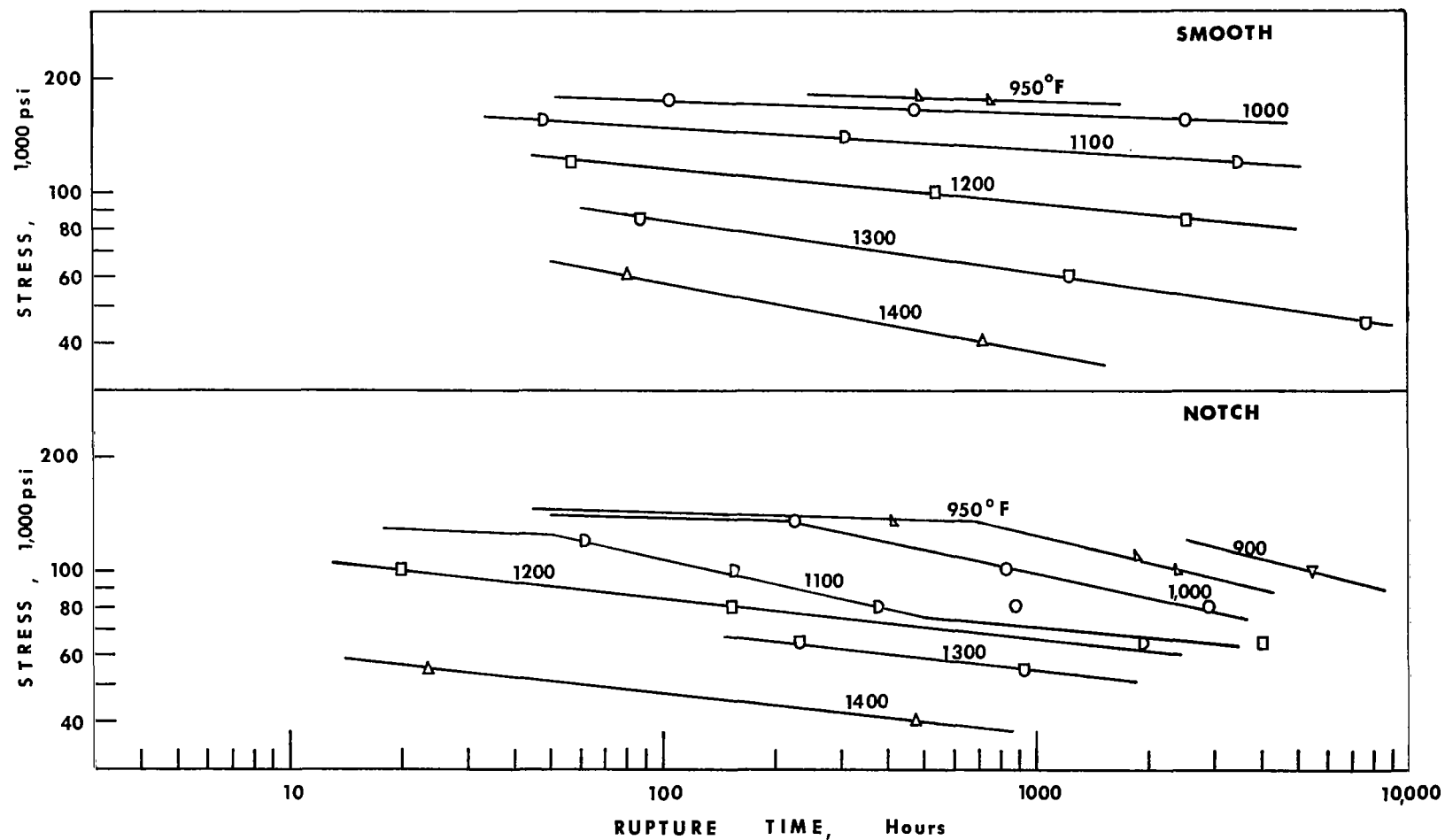


Figure 12. Stress versus rupture time data at temperatures from 900° to 1400°F obtained from smooth and notched specimens of 0.026-inch thick Waspaloy sheet solution treated 1/2 hour at 1825°F and aged 16 hours at 1400°F. Time-dependent notch sensitivity occurred for test temperatures from 900° to 1100°F but not from 1200° to 1400°F.

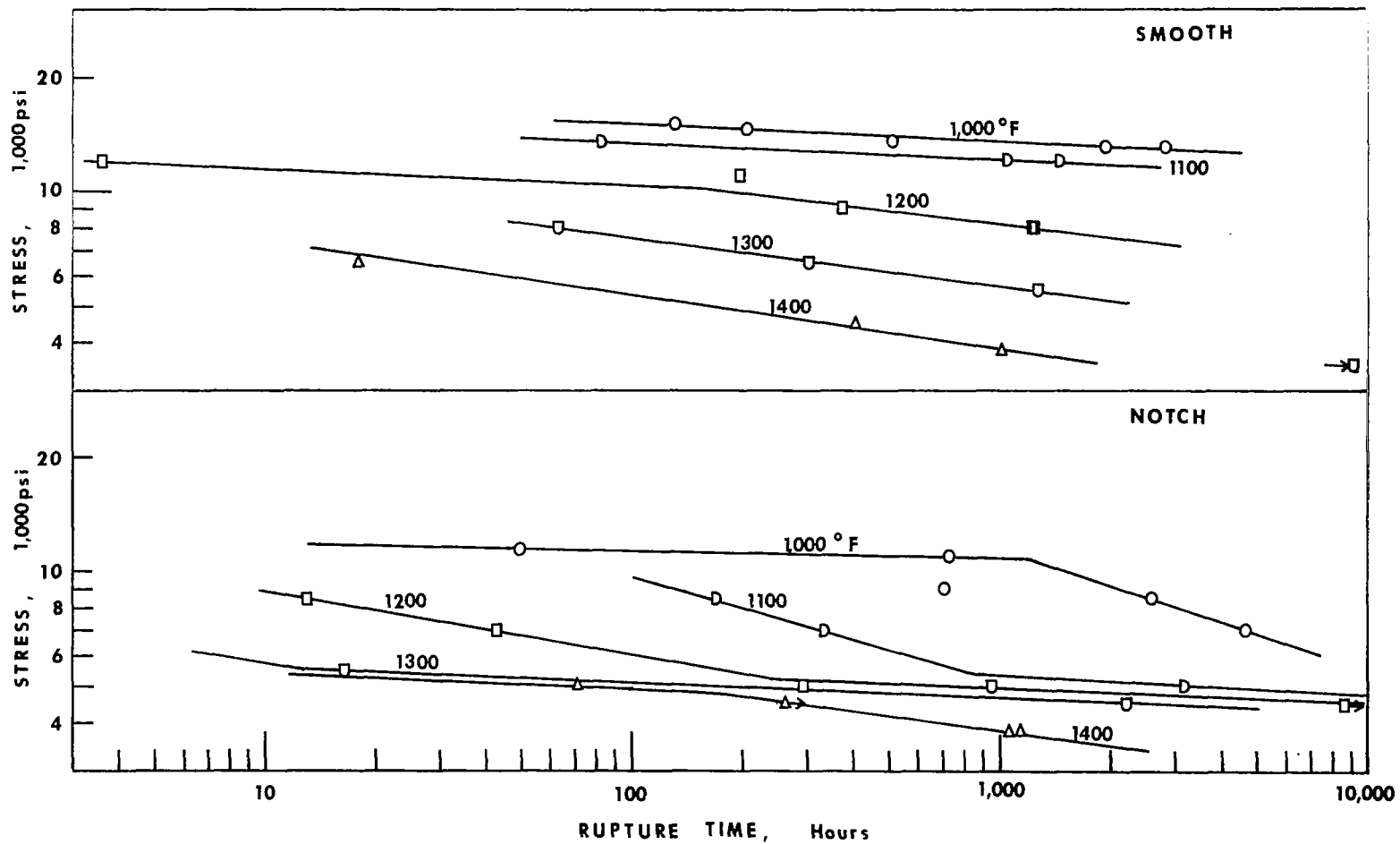


Figure 13. Stress versus rupture time data at temperatures from 1000° to 1400°F obtained from smooth and notched specimens of 0.026-inch thick Waspaloy sheet solution treated 1/2 hour at 1975°F and aged 16 hours at 1400°F. The time-dependent notch sensitivity is evident at test temperatures from 1000° to 1300°F, but does not occur at 1400°F.



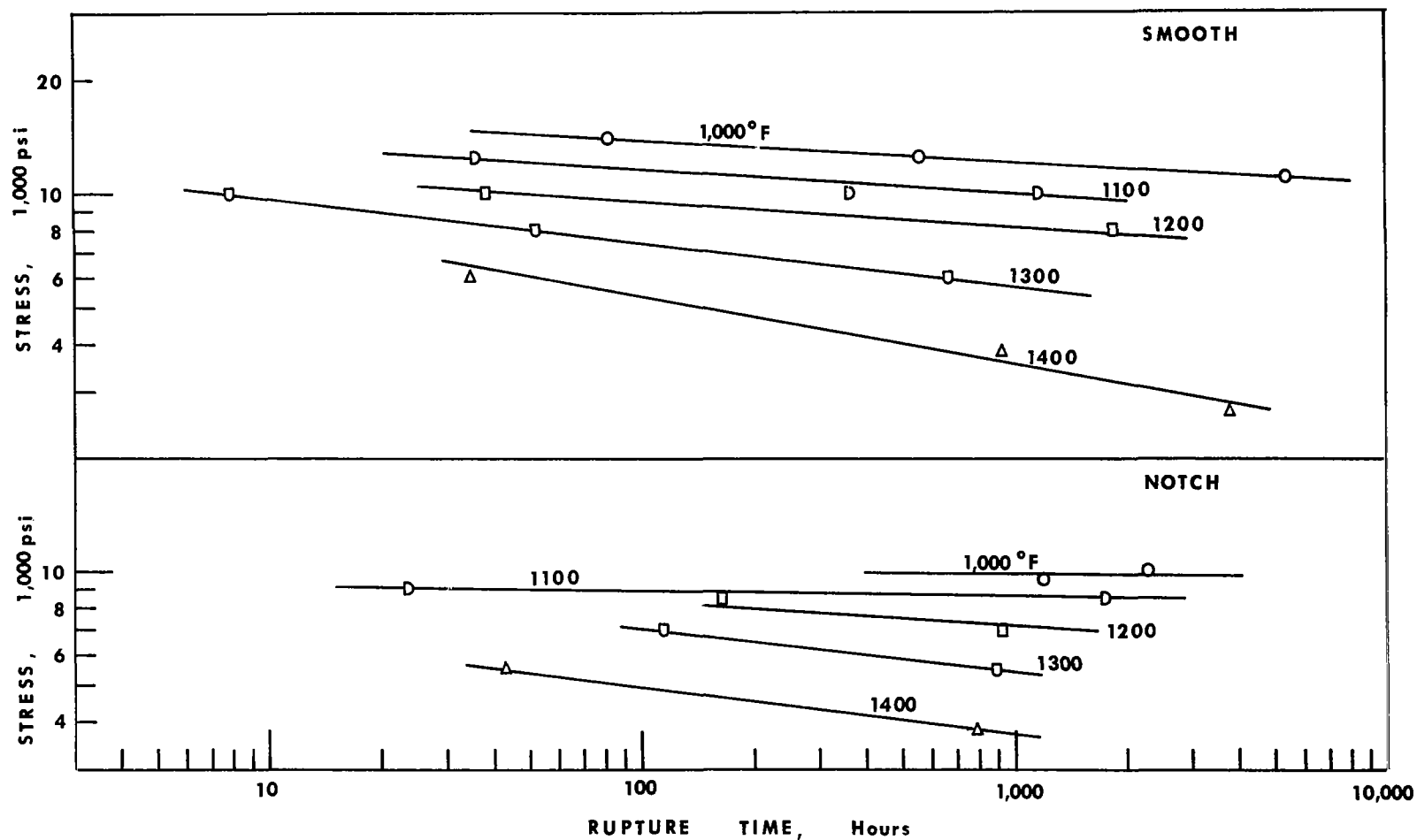


Figure 14. Stress versus rupture time data at temperatures from 1000° to 1400°F obtained from smooth and notched specimens of 0.026-inch thick Waspaloy sheet solution treated 1/2 hour at 1975°F and aged 10 hours at 1700°F. The tests show no time-dependent notch sensitivity at temperatures from 1000° to 1400°F.

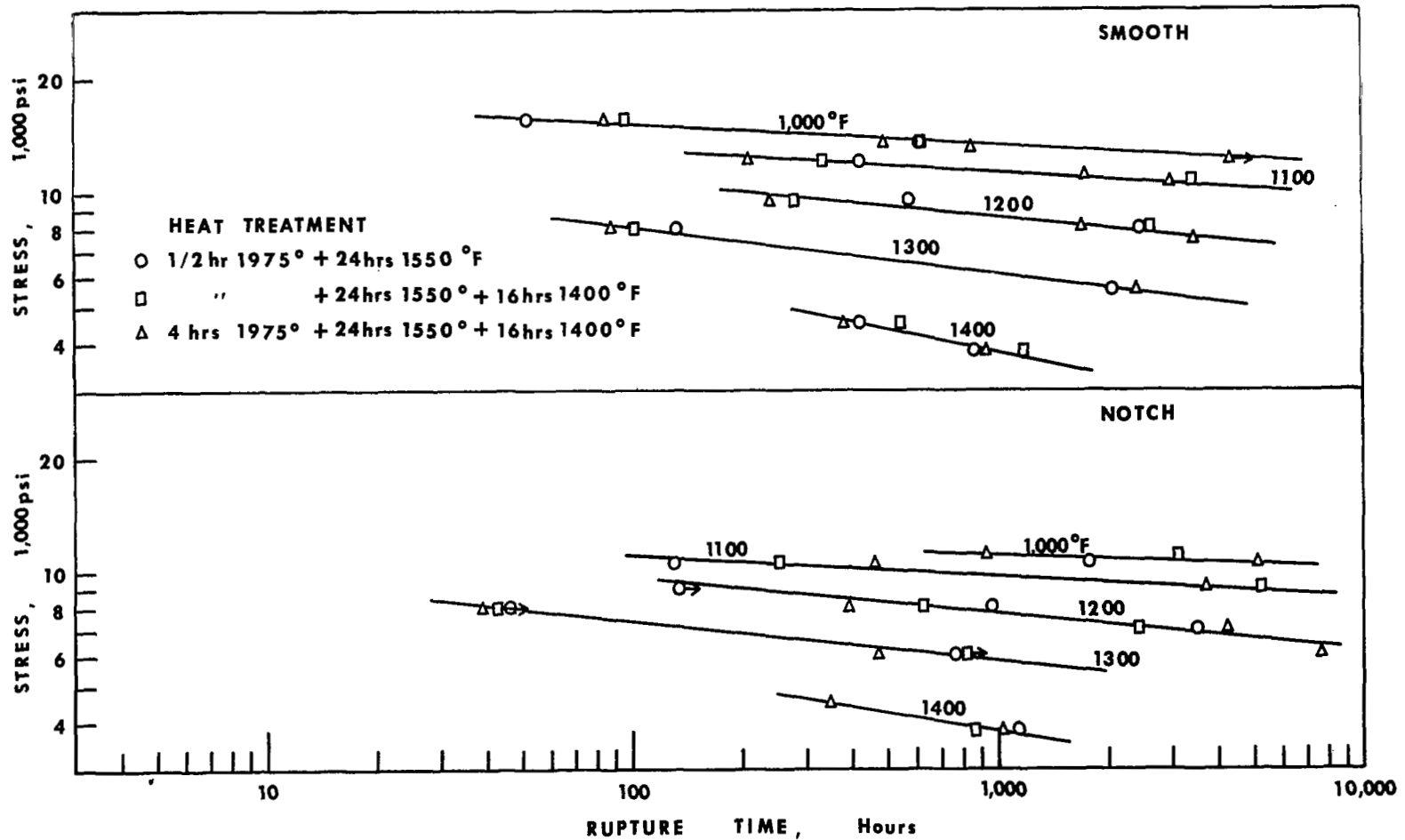


Figure 15. Stress versus rupture time data at temperatures from 1000° to 1400°F obtained from smooth and notched specimens of 0.026-inch thick Waspaloy sheet solution treated at 1975°F and aged. The tests show no time-dependent notch sensitivity at temperatures from 1000° to 1400°F with little difference in behavior for the heat treatment variations.

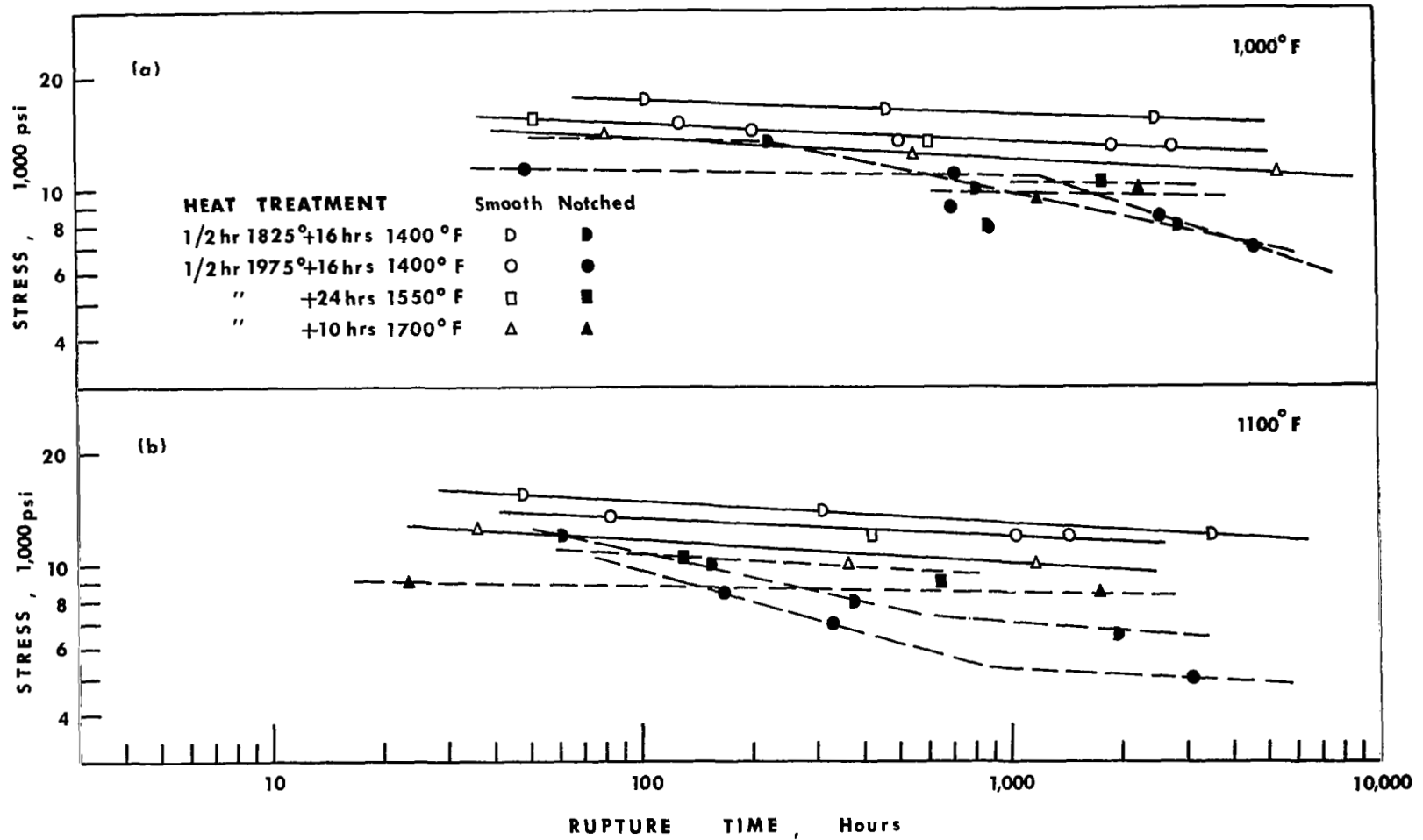


Figure 16. Stress versus rupture time data at temperatures from 1000° to 1400°F obtained from smooth and notched specimens of 0.026-inch thick Waspaloy sheet in the heat treated conditions.

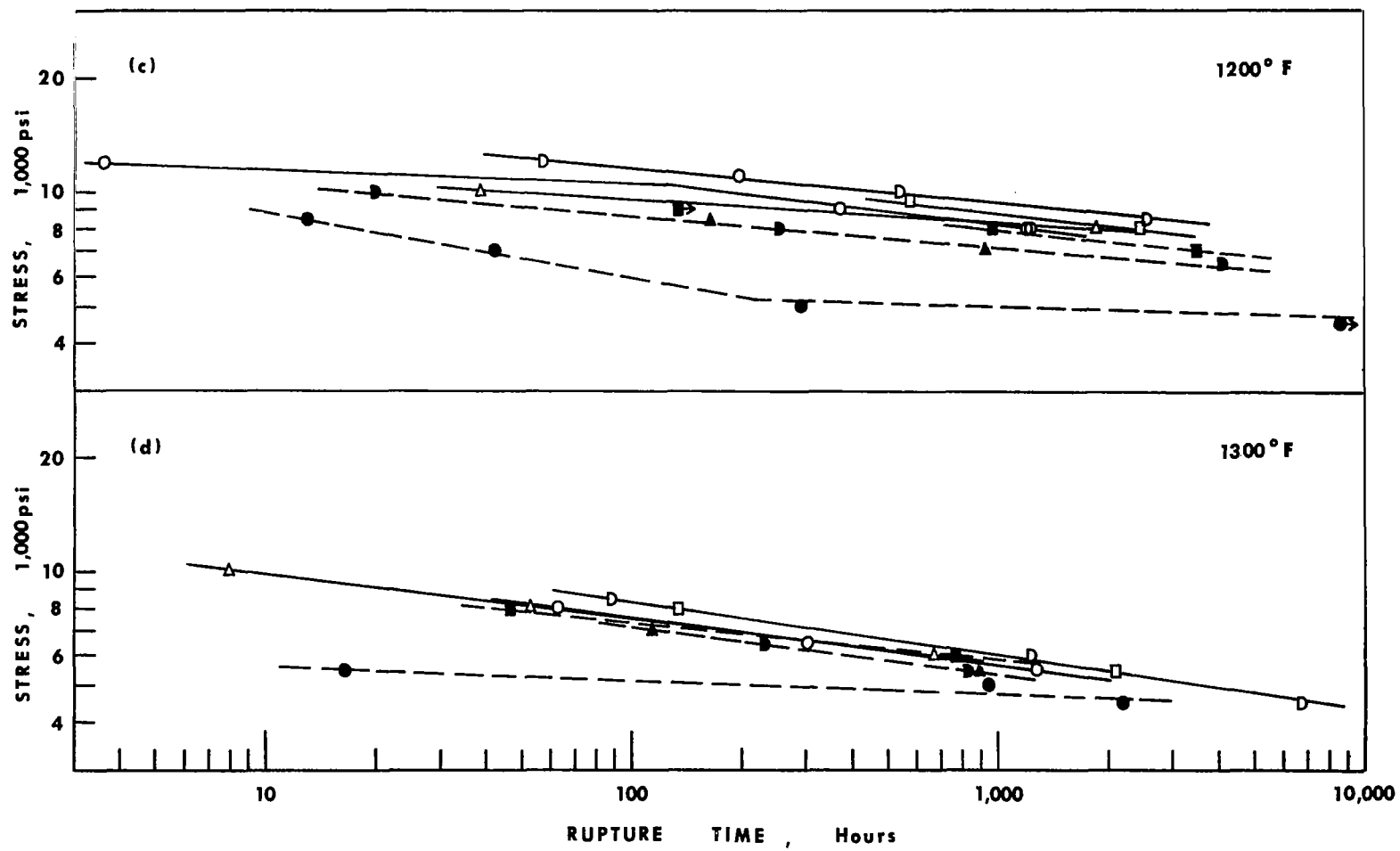


Figure 16 (Continued)

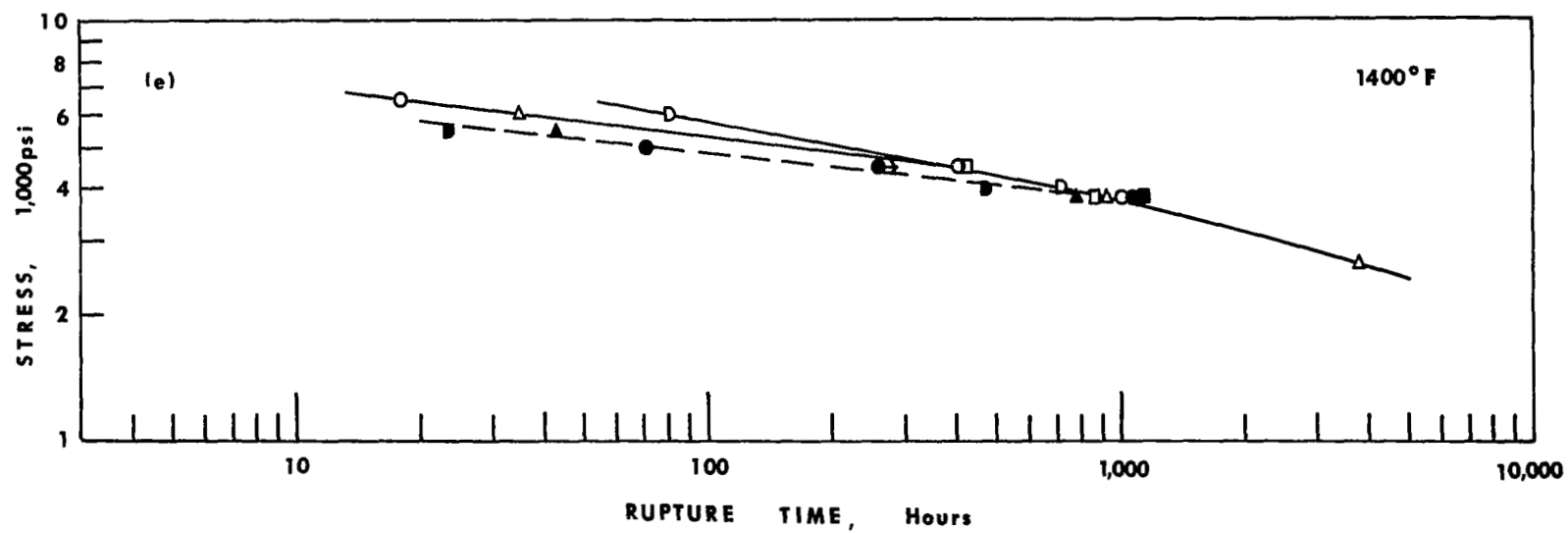


Figure 16 (Continued)

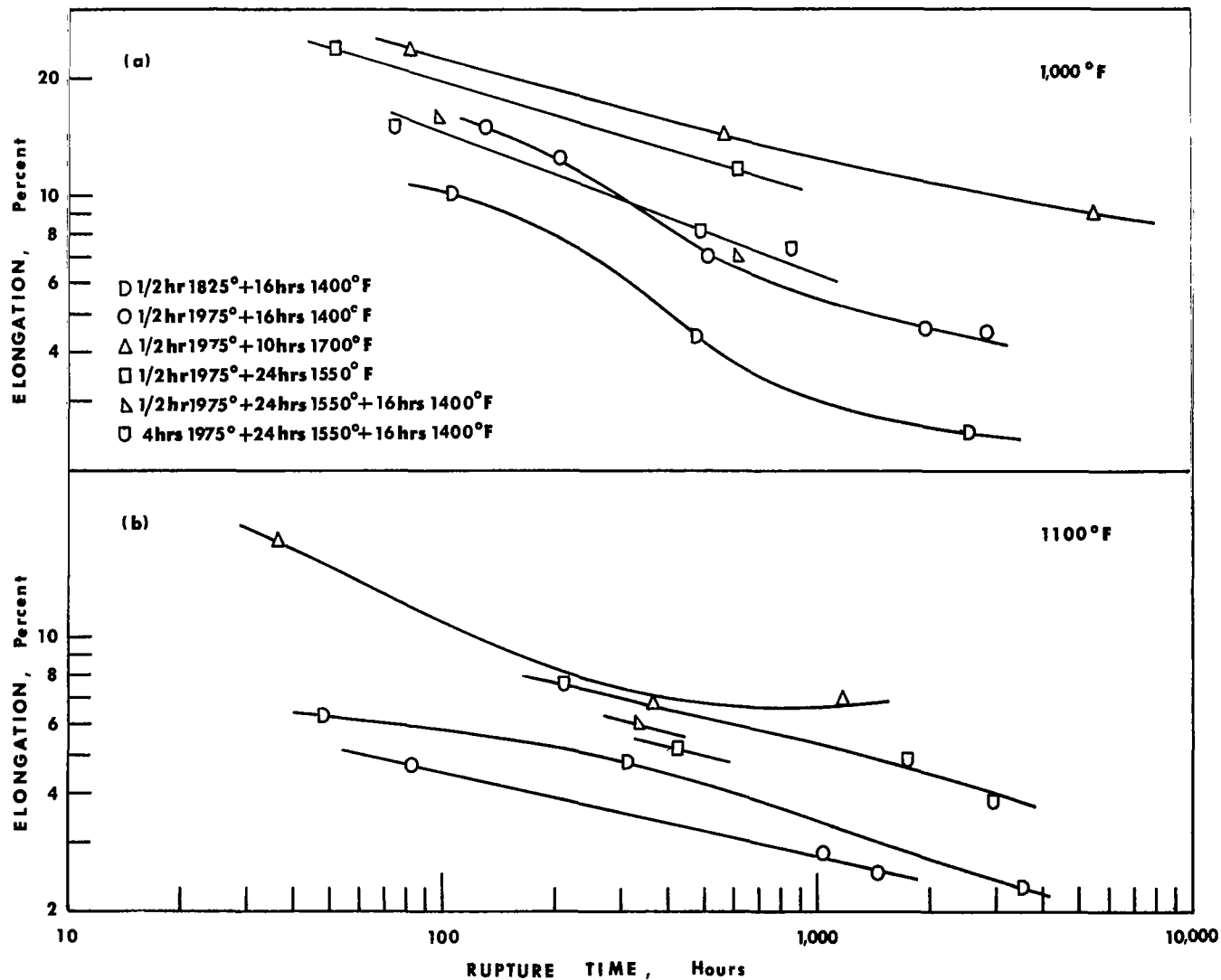


Figure 17. Elongation versus rupture time data at temperatures from 1000° to 1400°F for smooth specimens of 0.026-inch thick Waspaloy sheet in the heat treated condition.

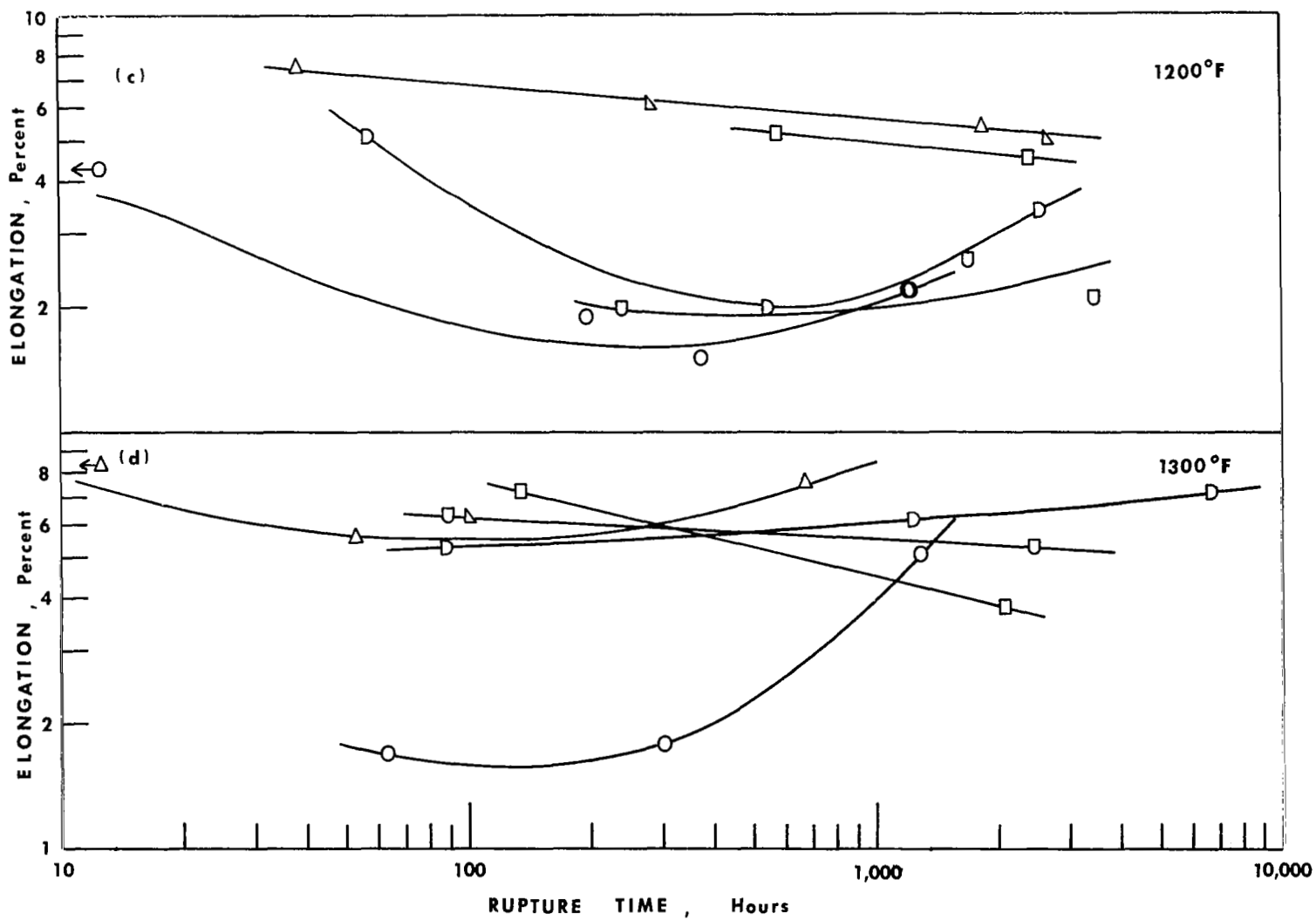


Figure 17 (Continued)

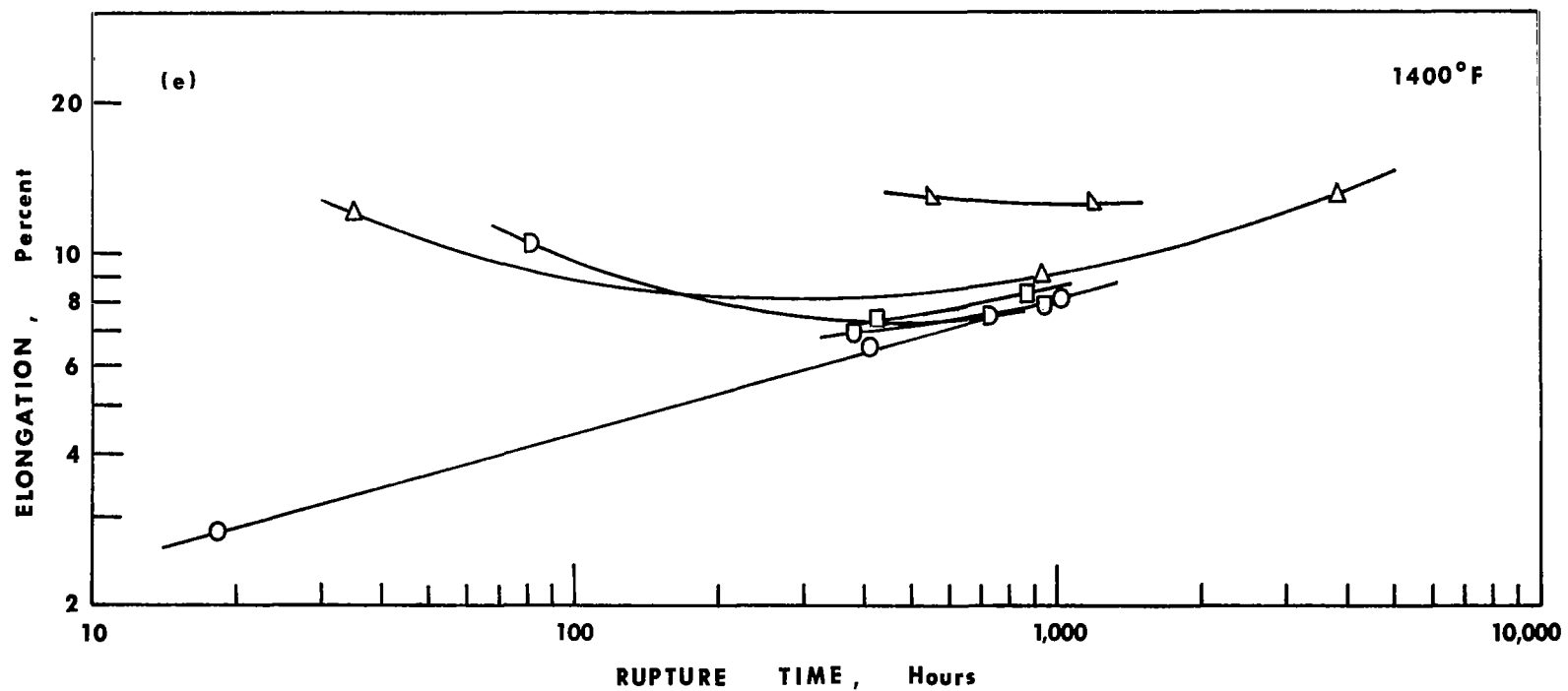
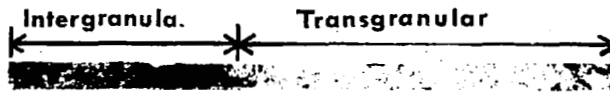


Figure 17 (Continued)





16X

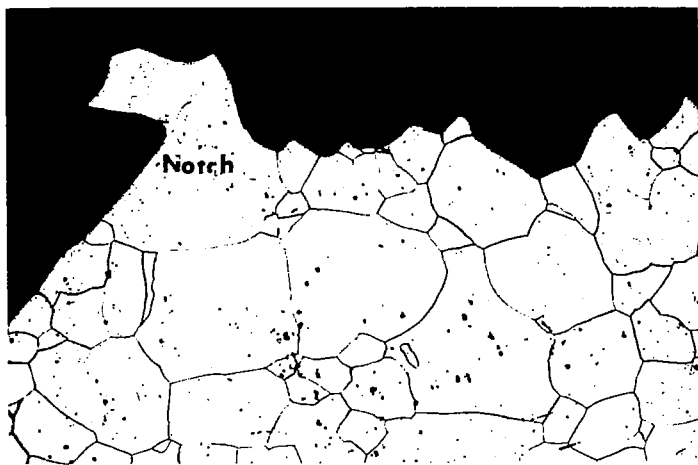
Smooth specimen of 0.026-inch thick Waspaloy sheet.  
(Material solution treated at 1825°F and aged at 1700°F;  
tested at 1200°F at 85 ksi; ruptured in 894.5 hours).



16X

Smooth specimen of 0.050-inch thick Waspaloy sheet.  
(Material solution treated at 1975°F and aged at 1400°F;  
tested at 1200°F at 90 ksi; ruptured in 513.1 hours).

Figure 18. Fracture surfaces of ruptured specimens. Failure of both smooth and notched creep-rupture specimens occurred by slow intergranular crack initiation and growth followed by rapid transgranular fracture.



(a)



(b)



(c)

Figure 19. Optical photomicrographs (50X) showing the fracture path across a specimen. (Notched specimen heat treated 1/2 hour at 2150°F plus 16 hours at 1400°F tested at 1200°F at 65 ksi, ruptured in 59.1 hours). Intergranular fracture is shown in (a) to (c). Deformation associated with the transgranular part of the fracture (d) through (f) is evident in (e).

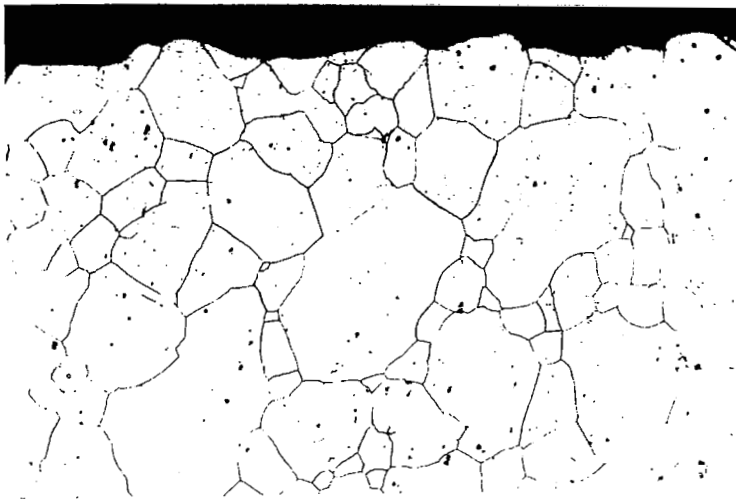


Figure 19 continued.

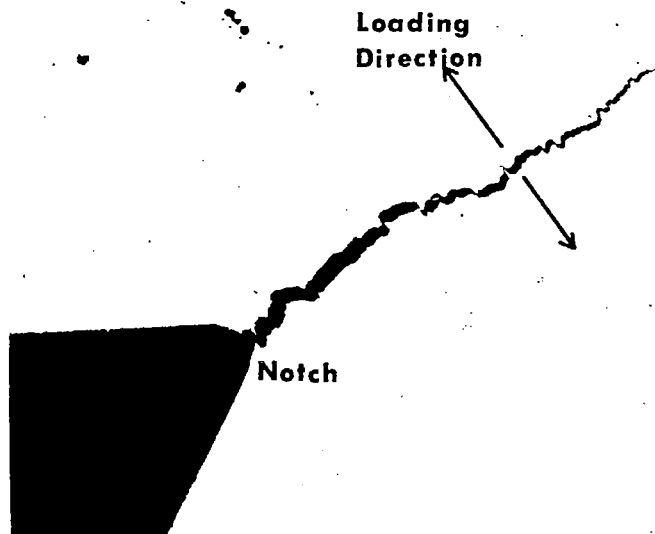
(d)



(e)

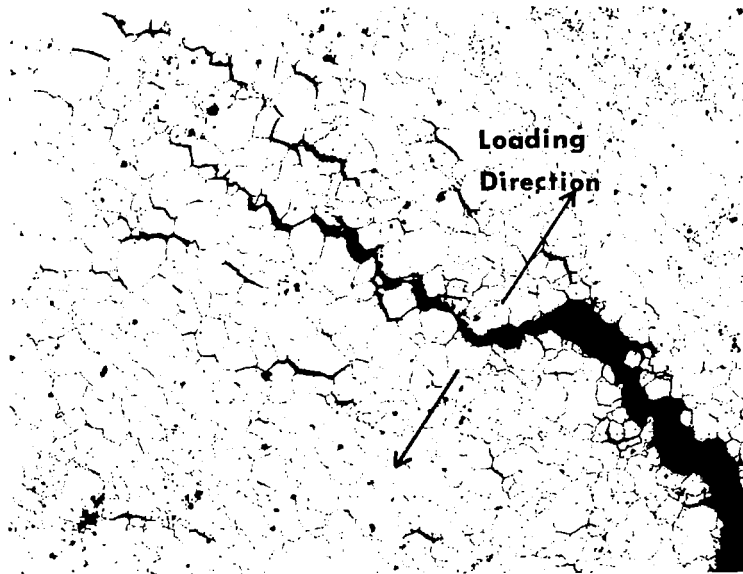


(f)



50X

Figure 20. Photomicrograph showing an intergranular crack developing from a machined notch ( $K_t=20$ ). (0.026-inch thick Waspaloy sheet solution treated at 1900°F and aged at 1700°F; tested at 1000°F at 85 ksi; discontinued after 6147 hours.)



100X

Figure 21. Photomicrograph showing subsidiary cracking in the region of the major intergranular crack. (0.026-inch thick Waspaloy sheet notched specimen solution treated at 1975°F and aged at 1400°F; tested at 1400°F at 45 ksi; ruptured in 260 hours in the pin holes.)

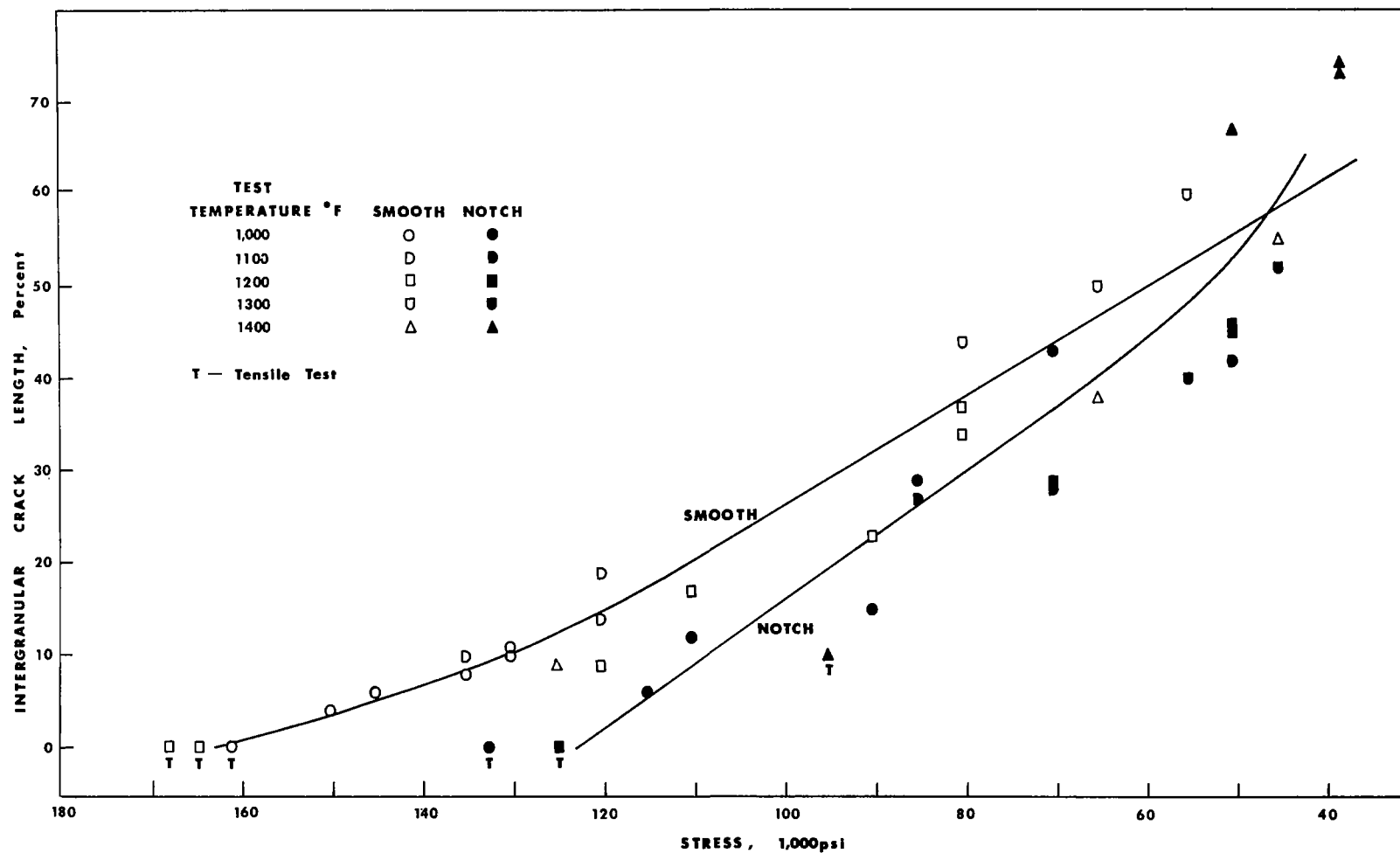


Figure 22. Intergranular crack length versus initial loading stress for Waspaloy solution treated 1/2 hour at 1975°F and aged 16 hours at 1400°F. The data was obtained from notched and smooth fractured specimens tensile and rupture tested at temperatures from 1000° to 1400°F. For a given test stress, the intergranular crack lengths in smooth and notched specimens differ to some extent, particularly at the higher stresses.

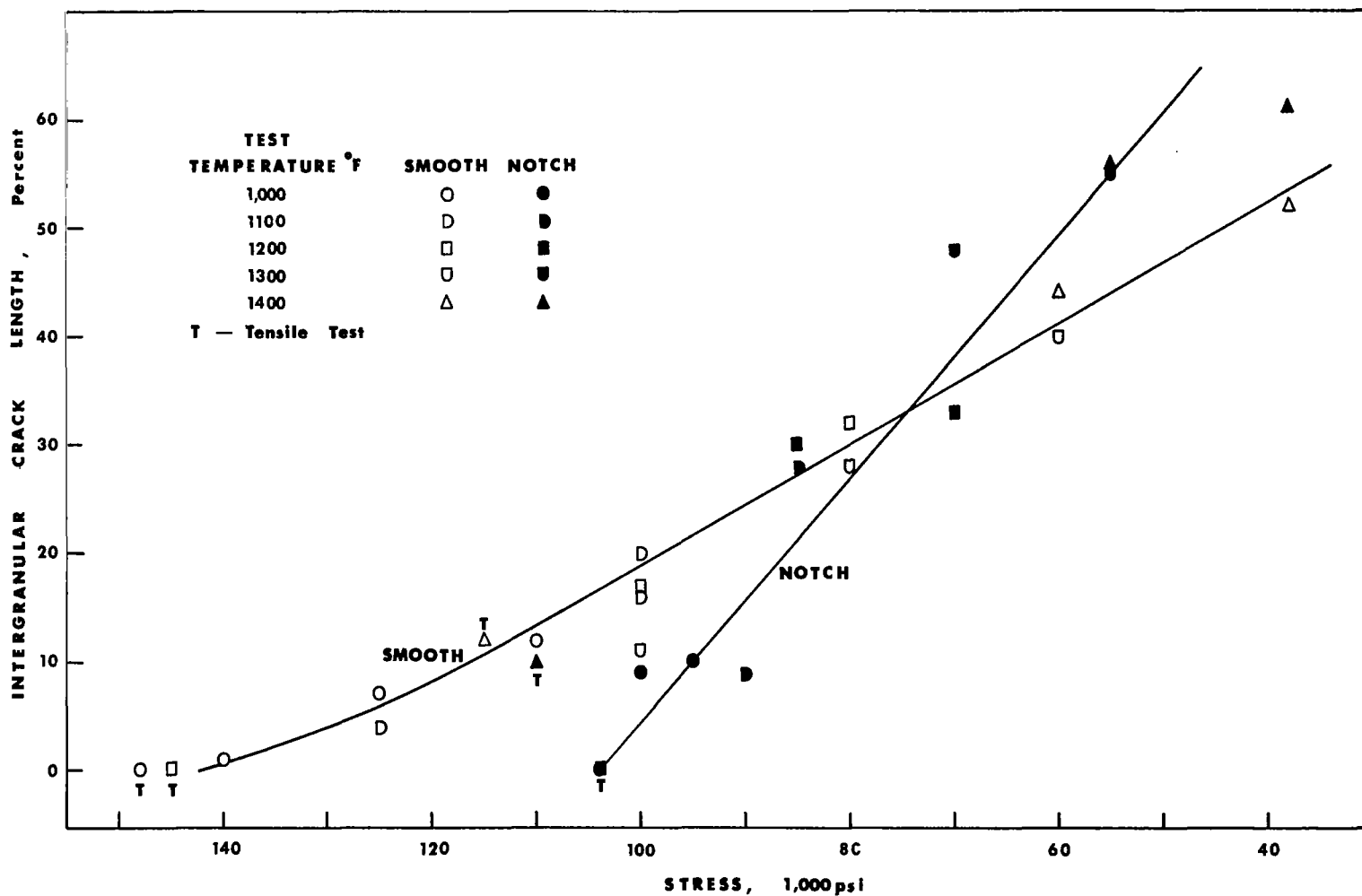


Figure 23. Intergranular crack length versus initial loading stress for Waspaloy solution treated 1/2 hour at 1975°F and aged 10 hours at 1700°F. The data was obtained from notched and smooth fractured specimens tensile and rupture tested at temperatures from 1000° to 1400°F. For the majority of test stresses the intergranular crack lengths for smooth and notched specimens differ to some degree.

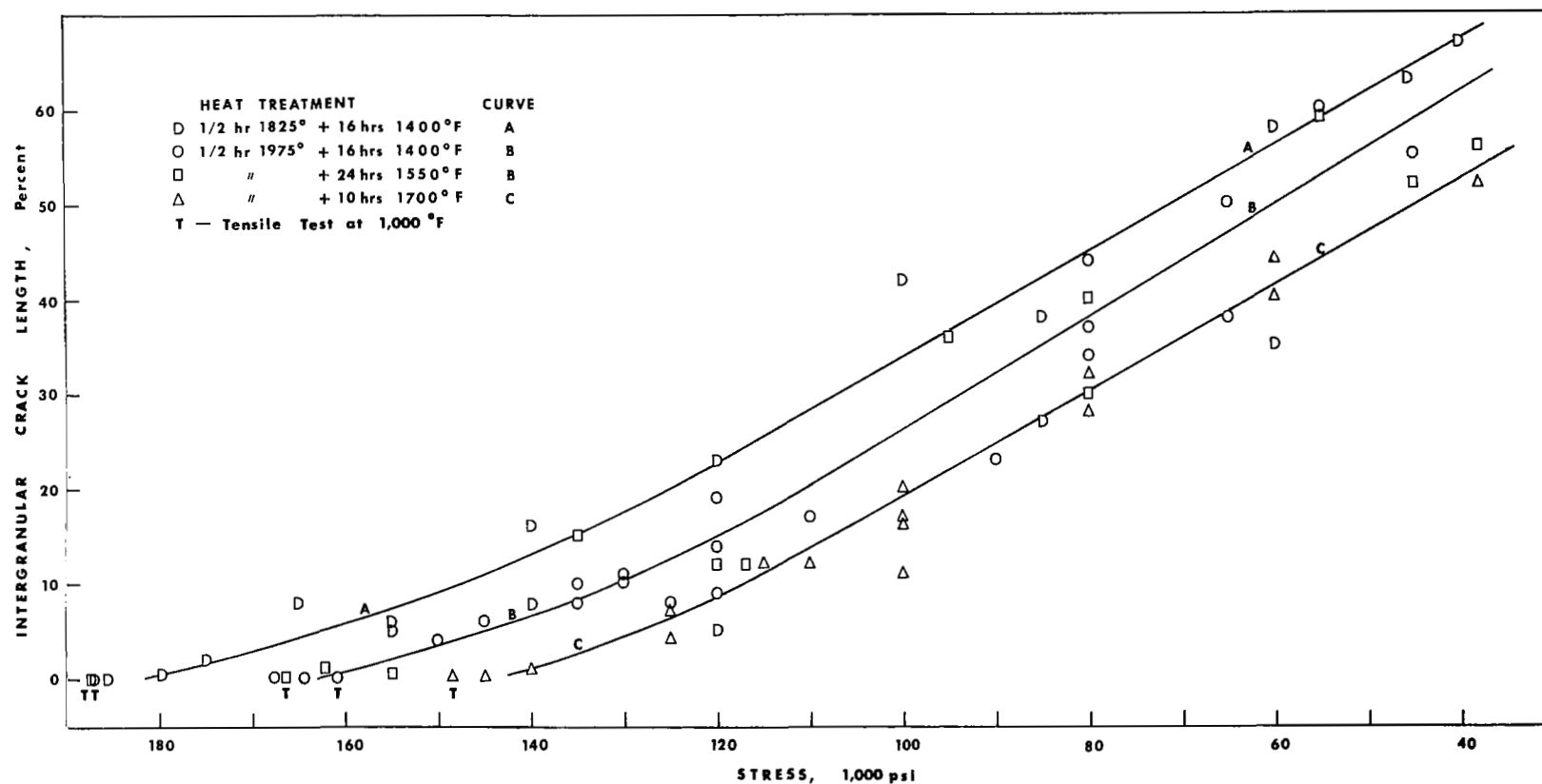


Figure 24. Intergranular crack length versus loading stress for Waspaloy sheet materials in the heat treated condition. The data were obtained from the fractures of smooth specimens tensile and rupture tested at temperatures from 1000° to 1400°F. For a given stress the intergranular crack length increased with the transgranular strength, as indicated by the tensile tests at 1000°F (zero intergranular crack length).

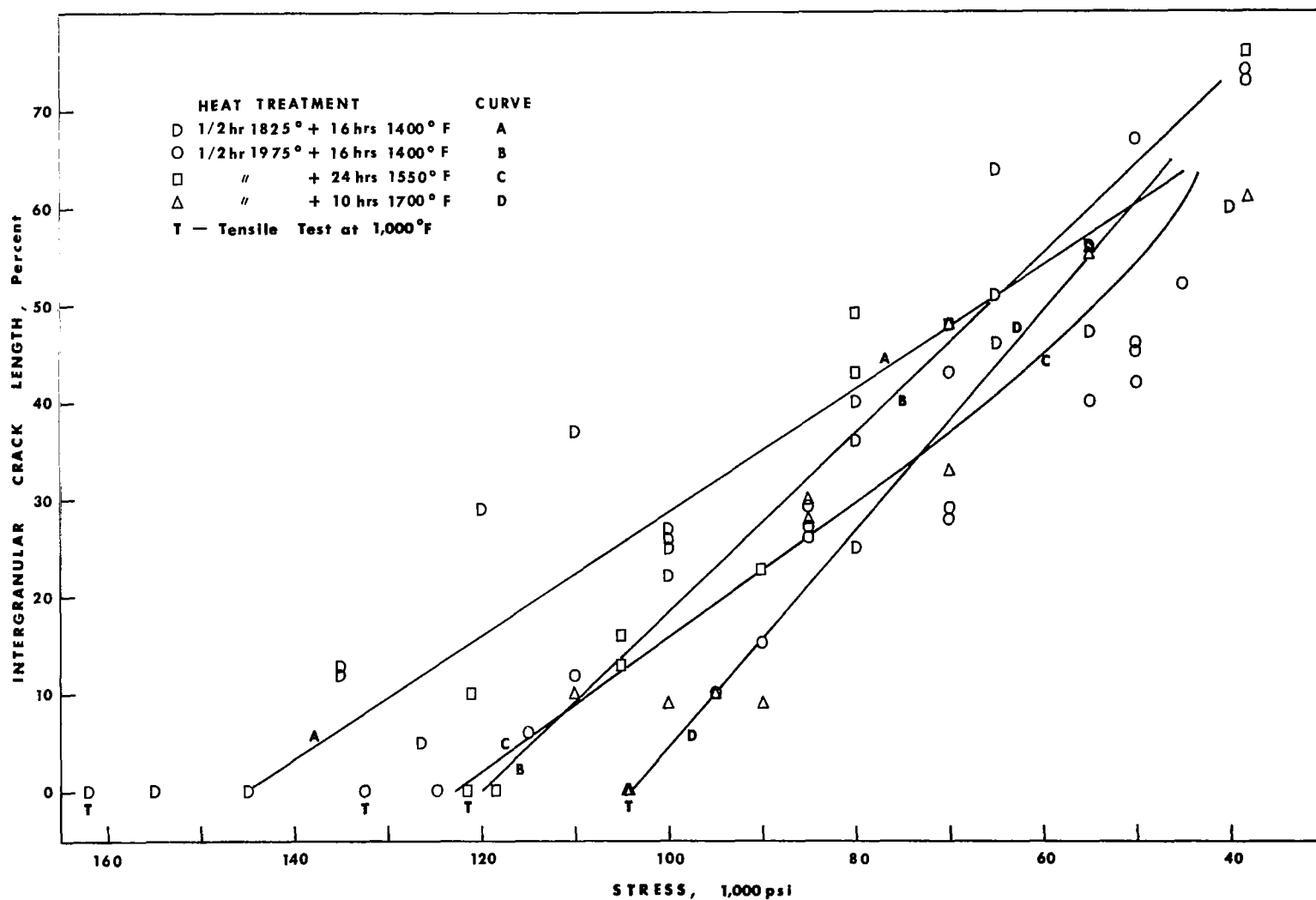


Figure 25. Intergranular crack length versus loading stress for Waspaloy sheet materials in the heat treated conditions. The data were obtained from the fractures of notched specimens tensile and rupture tested at temperatures from 1000° to 1400°F.



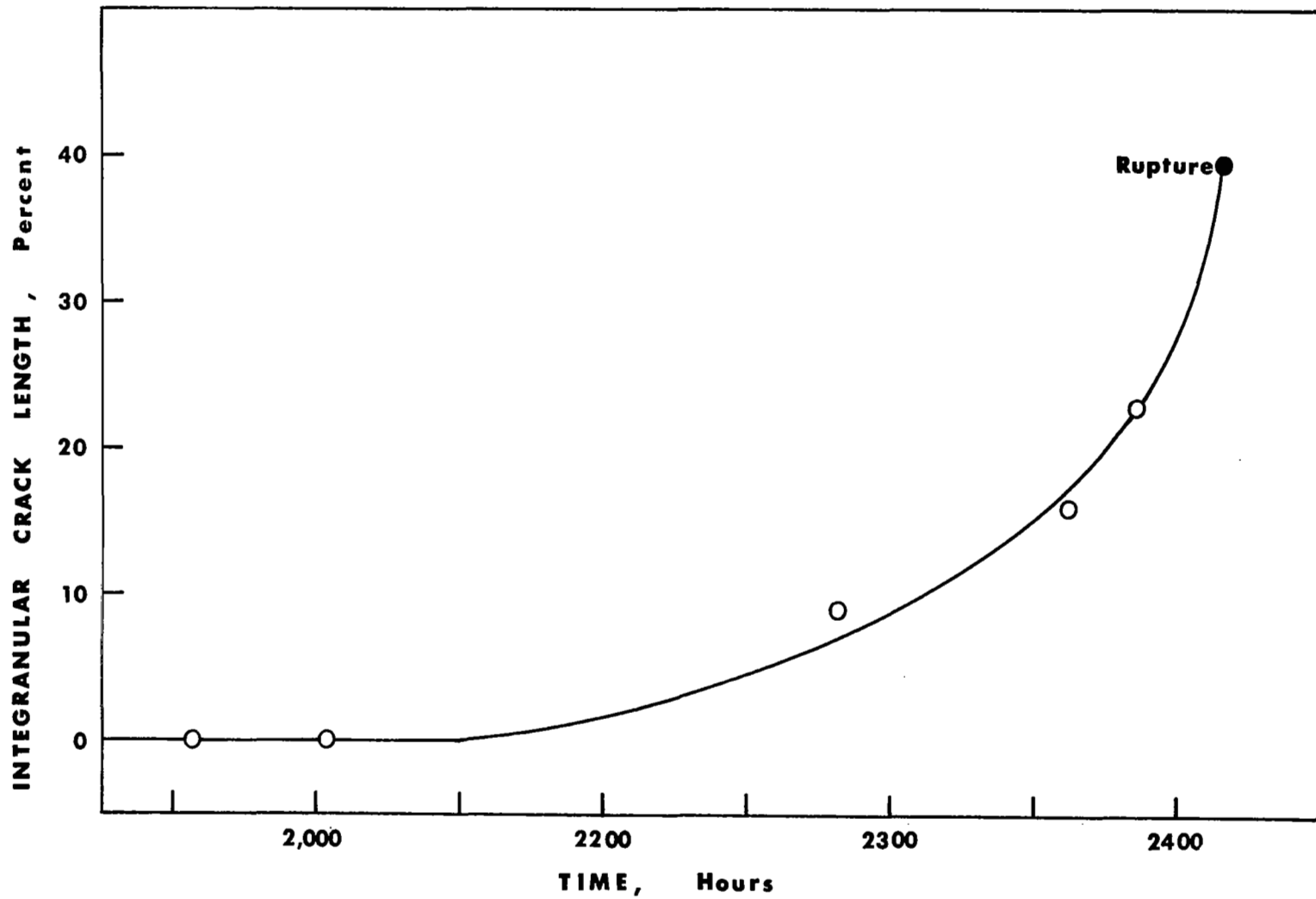
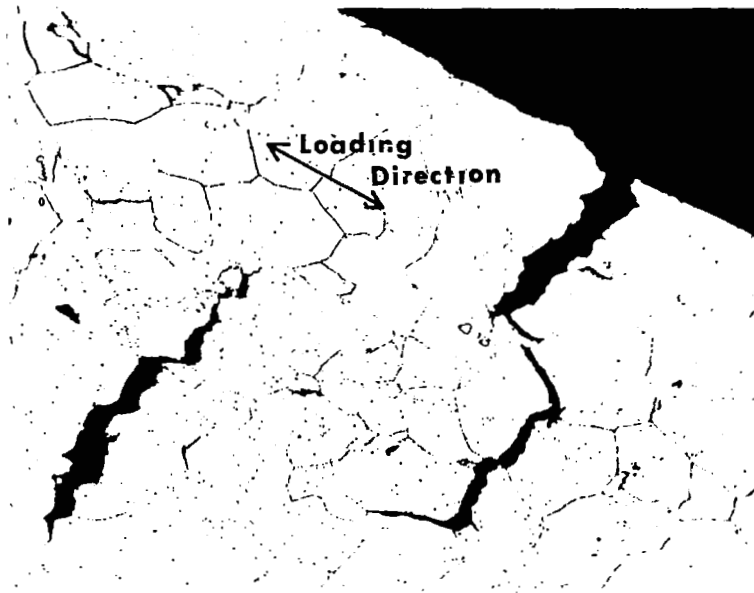
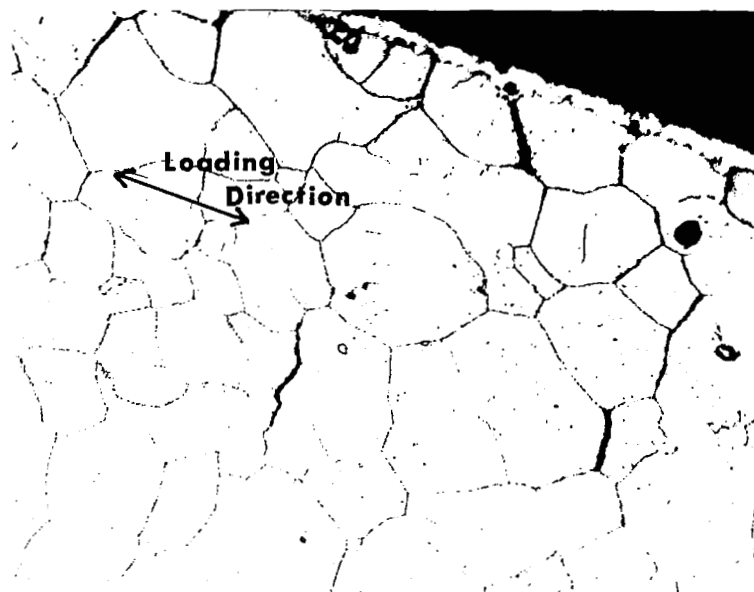


Figure 26. Intergranular crack length versus time for a notched specimen of Waspaloy heat treated 1/2 hour at 1975°F plus 16 hours at 1400°F and tested at 1000°F at 85 ksi. The crack growth rate increases with time which reflects the increase in stress on the load bearing section.



500X

Figure 27. Photomicrograph showing subsidiary cracking in a smooth specimen. (0.026-inch thick Waspaloy sheet solution treated at 1975°F and aged at 1400°F; tensile tested at 1200°F.)



500X

Figure 28. Photomicrograph showing subsidiary cracking in a smooth specimen. (0.026-inch thick Waspaloy sheet solution treated at 1975°F and aged at 1700°F; tested at 1200°F at 80 ksi; ruptured in 1,870 hours.)

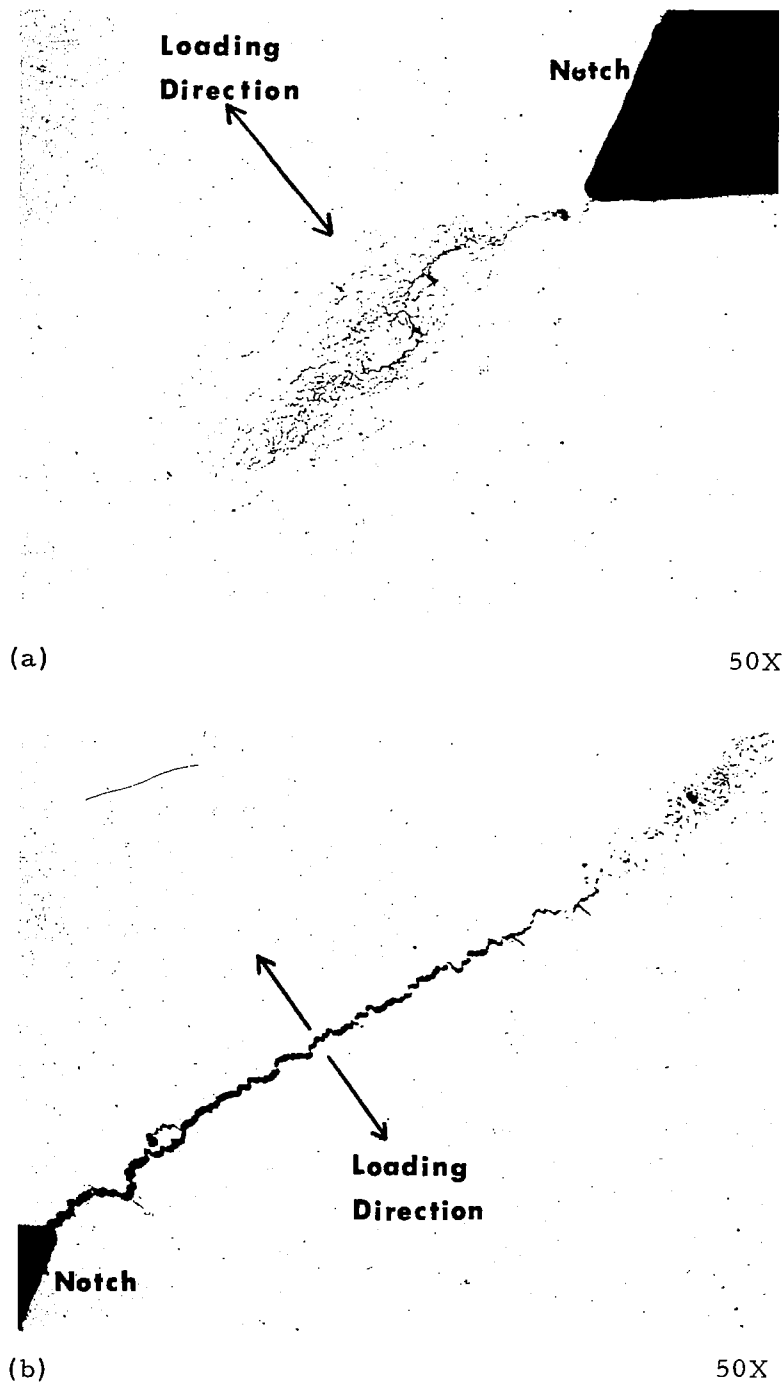


Figure 29. Optical photomicrographs of a notched specimen of Waspaloy polished to remove the surface oxidized layer. (Heat treated 1/2 hour at 1975°F plus 16 hours at 1400°F, exposed for 90 percent of its rupture life at 1000°F at 80 ksi). Microcracks at the base of the notch in (a) are an early stage of crack initiation. At the other notch, a through the thickness crack is evident, (b) a later stage of intergranular crack growth.

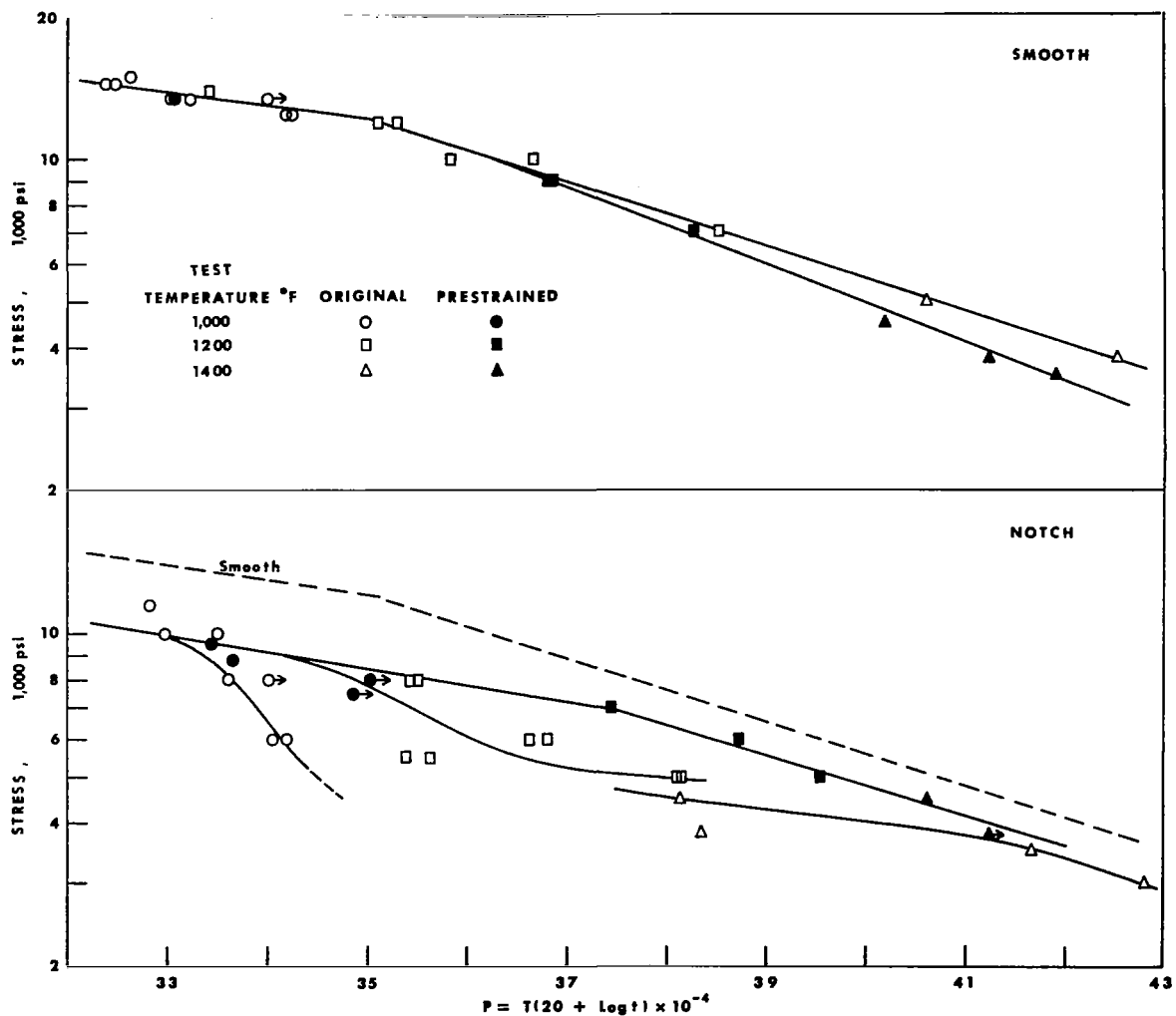


Figure 30. Time-temperature dependence of the rupture strengths of smooth and notched specimens of 0.026-inch thick Waspaloy sheet annealed and aged 16 hours at 1400°F. Prestraining of smooth specimens small amounts at the test temperature reduced the strengths to a limited extent. For notched specimens prestraining by loading to 115 ksi at 1000°F prior to testing, eliminated the time-dependent notch sensitivity.

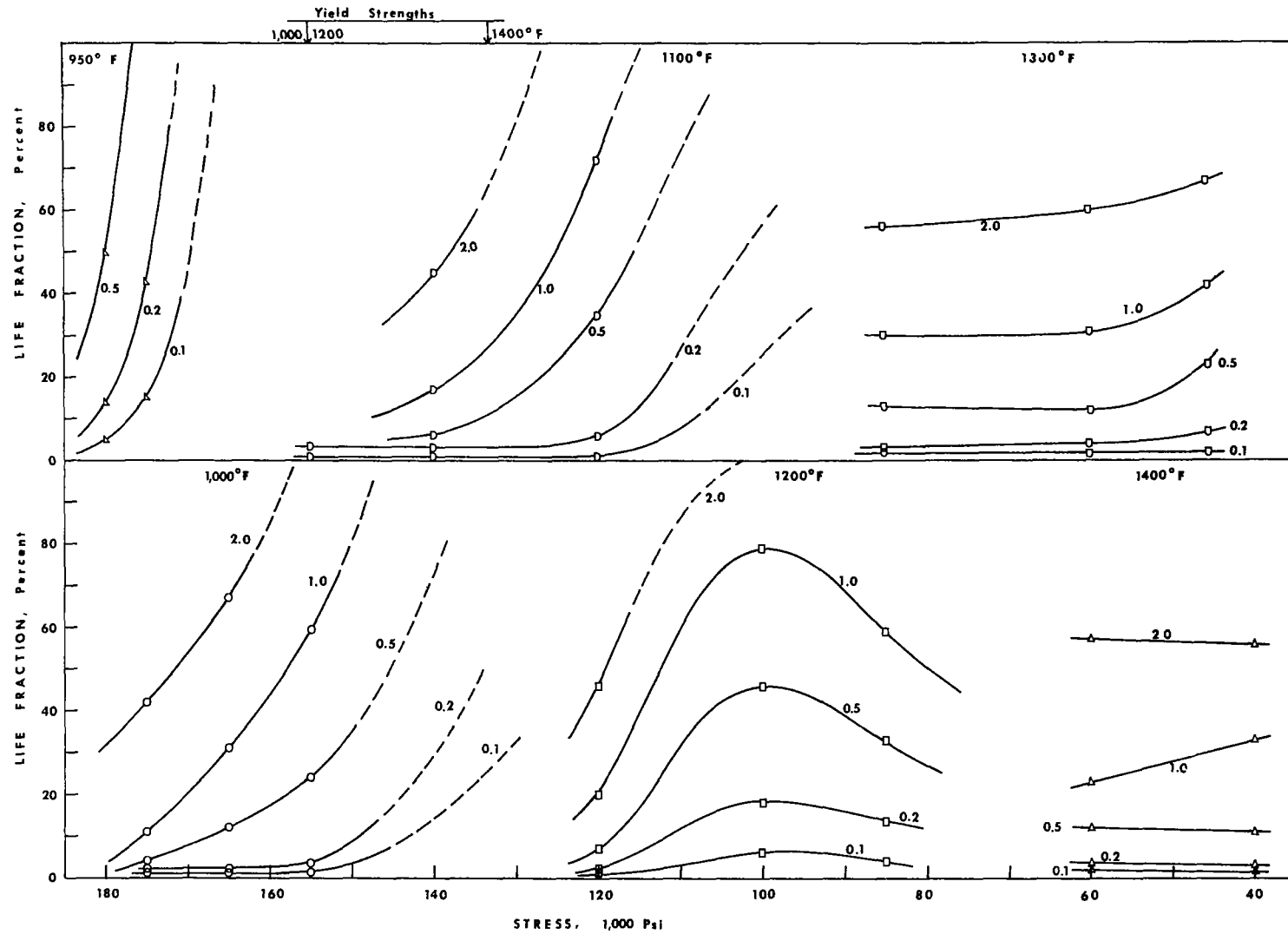


Figure 31. Iso-creep strain curves of life fraction versus stress at temperatures from 950° to 1400°F for 0.026-inch thick Waspaloy sheet solution treated 1/2 hour at 1825°F and aged 16 hours at 1400°F. Time-dependent notch sensitivity occurred under test conditions where (i) large amounts of rupture life were utilized for small amounts of creep at test temperatures 950°, 1000° and 1100°F and (ii) the test stresses were below the approximate 0.2 percent offset yield stress.

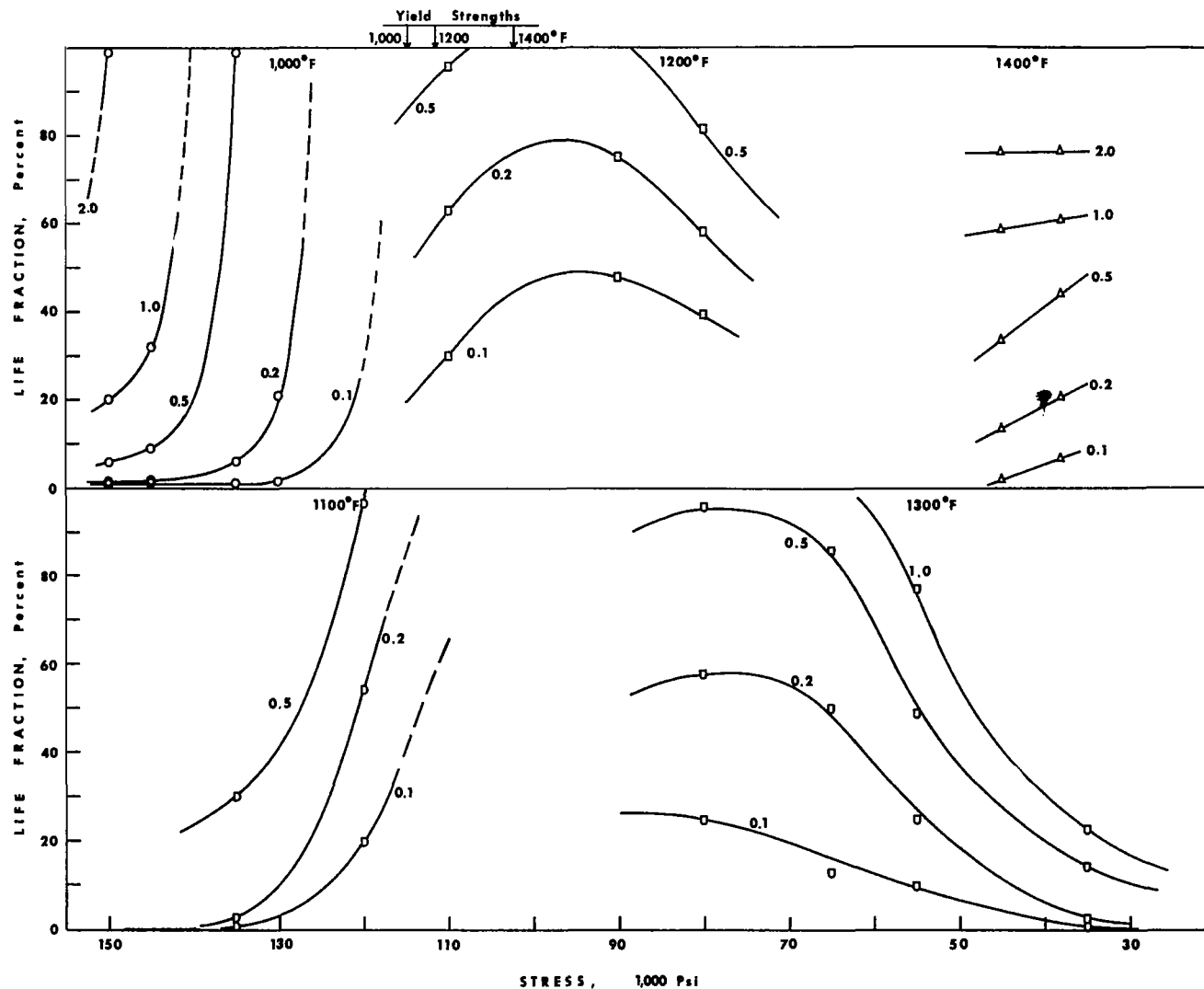


Figure 32. Iso-creep strain curves of life fraction versus stress at temperatures from 1000° to 1400°F for 0.026-inch thick Waspaloy sheet solution treated 1/2 hour at 1975°F and aged 16 hours at 1400°F. Time-dependent notch sensitivity occurred under test conditions: (i) large amounts of rupture life were utilized for small amounts of creep at temperatures from 1000° to 1300°F and (ii) the test stresses were below the approximate 0.2 percent offset yield stress.

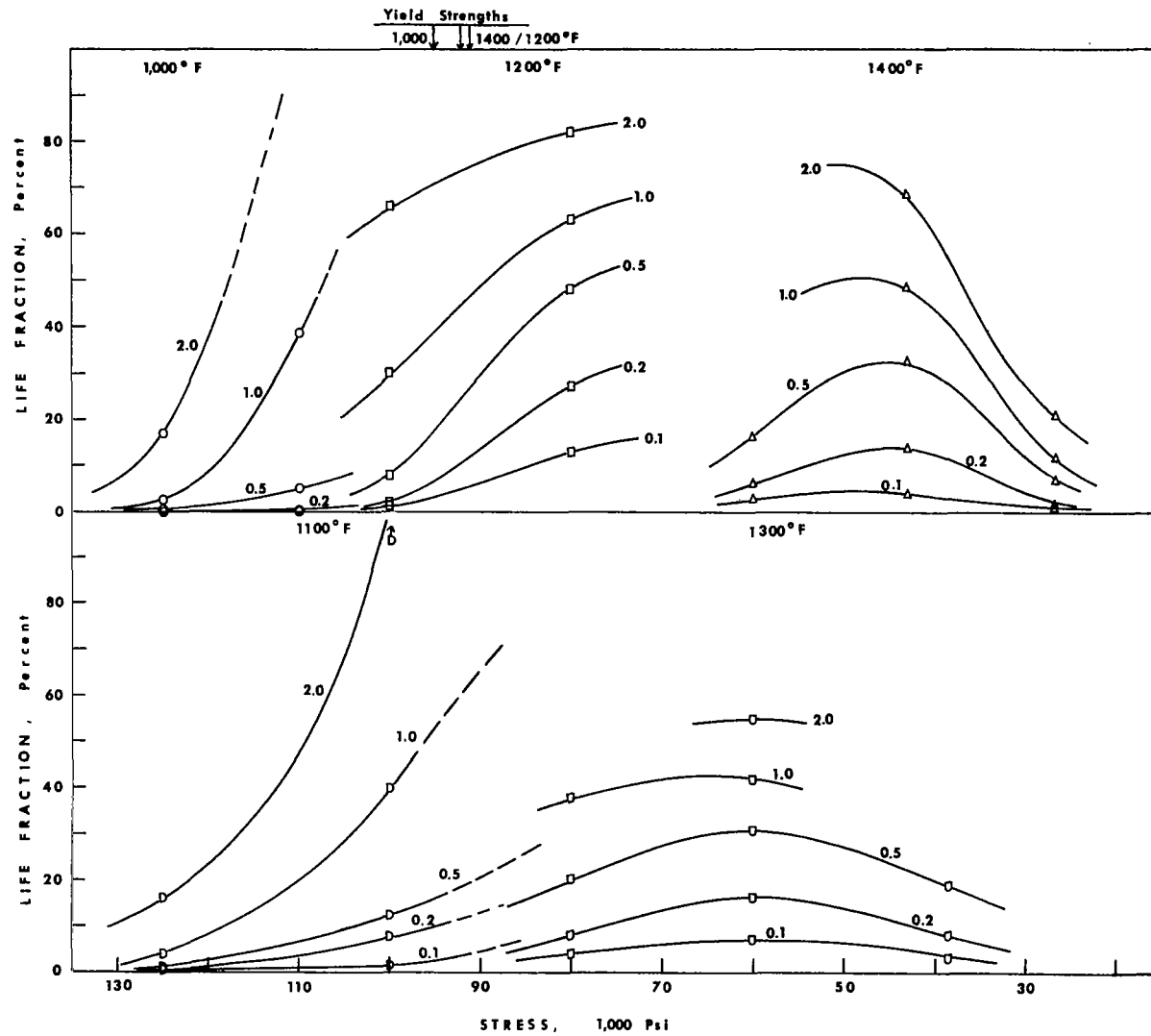


Figure 33. Iso-creep strain curves of life fraction versus stress at temperatures from 1000° to 1400°F for 0.026-inch thick Waspaloy sheet heat treated 1/2 hour at 1975°F and aged 10 hours at 1700°F. For the test conditions evaluated the life fraction utilized for small amounts of creep was relatively low and no time-dependent notch sensitivity was observed.

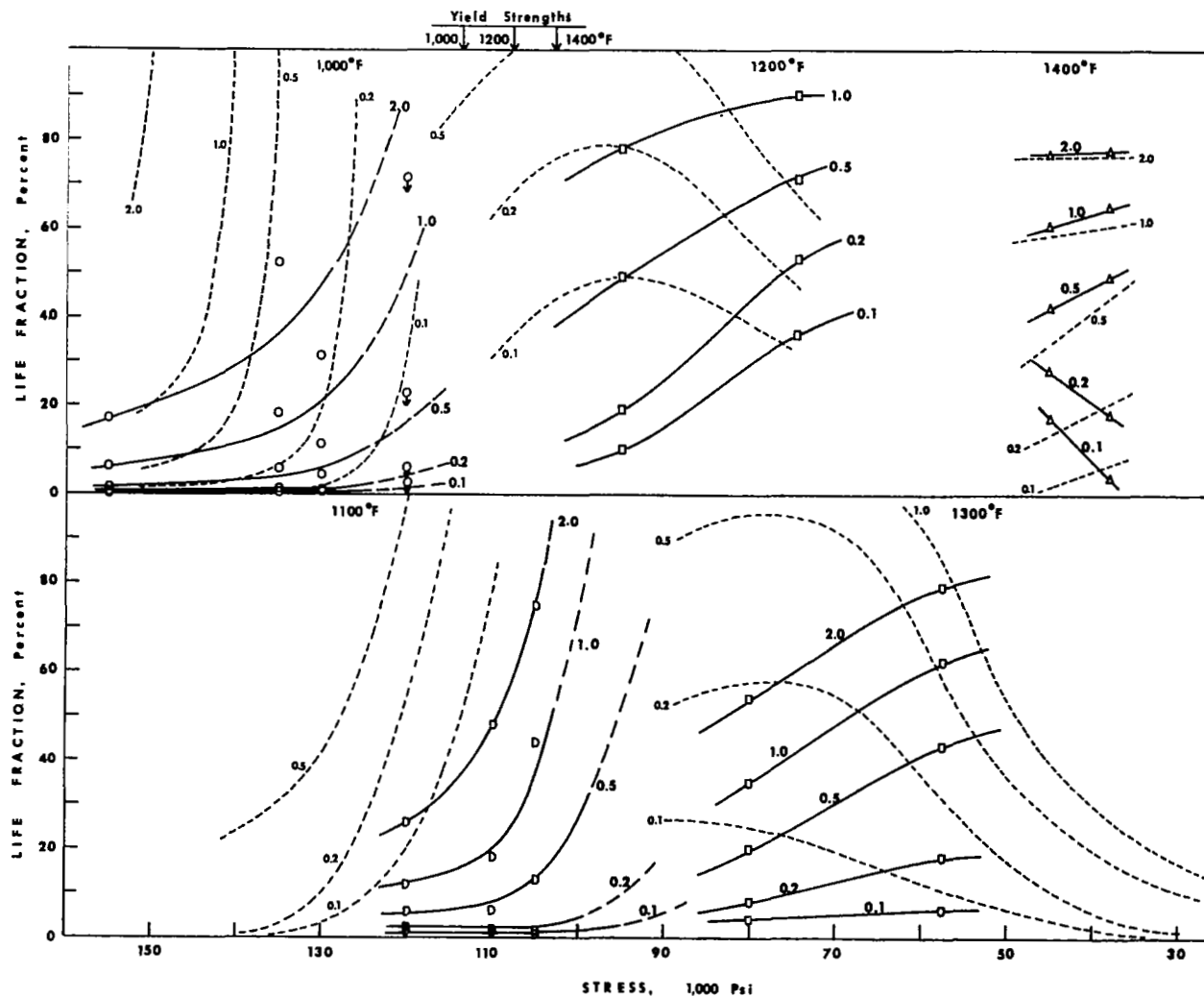
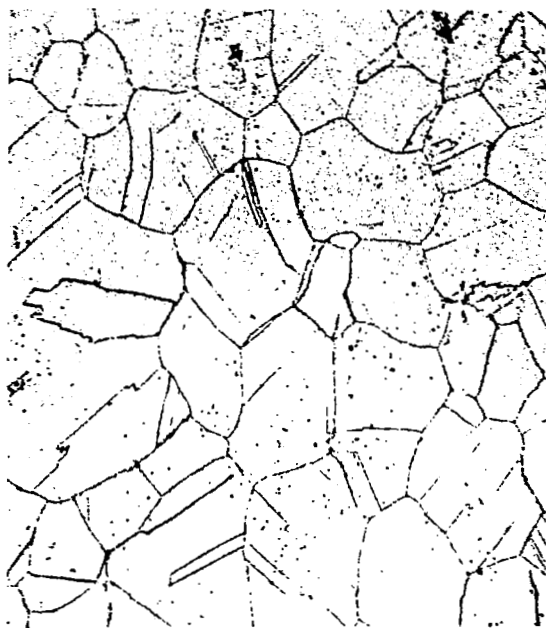


Figure 34. Iso-creep strain curves of life fraction versus stress at temperatures from 1000° to 1400°F for 0.026-inch thick Waspaloy sheet solution treated 4 hours at 1975°F and aged 24 hours at 1550°F plus 16 hours at 1400°F. The dashed lines are the curves for the material heat treated 1/2 hour at 1975°F and aged 16 hours at 1400°F (see Figure 32). The two heat treated materials exhibited similar smooth specimen strengths. However, while aging at 1400°F resulted in time-dependent notch sensitivity, none was observed after aging at 1550°F. This can be attributed to the lower life fractions for the latter material.





(a) 500X

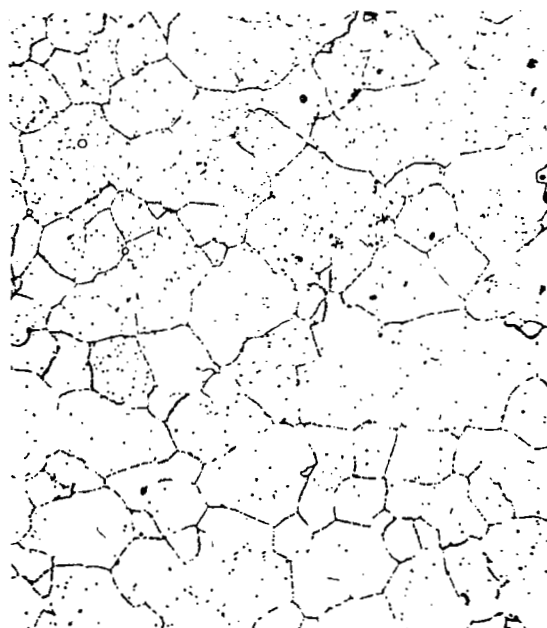
Aged 16 hours at 1400°F

Solution Treated 1/2-hour at 1825°F



(b) 500X

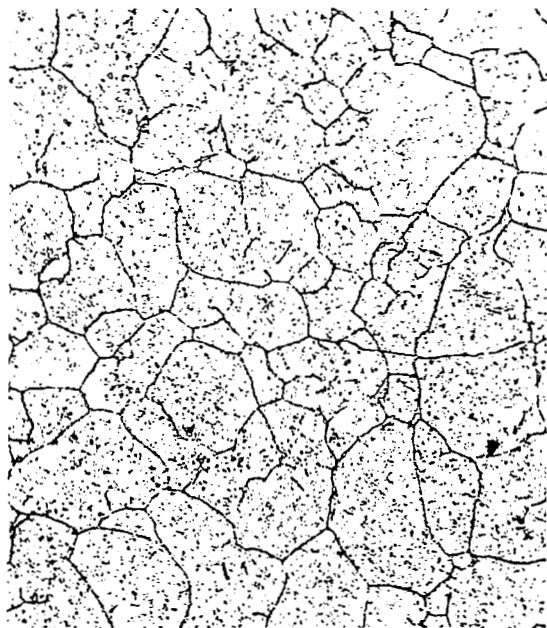
Aged 10 hours at 1700°F



(c) 500X

Aged 16 hours at 1400°F

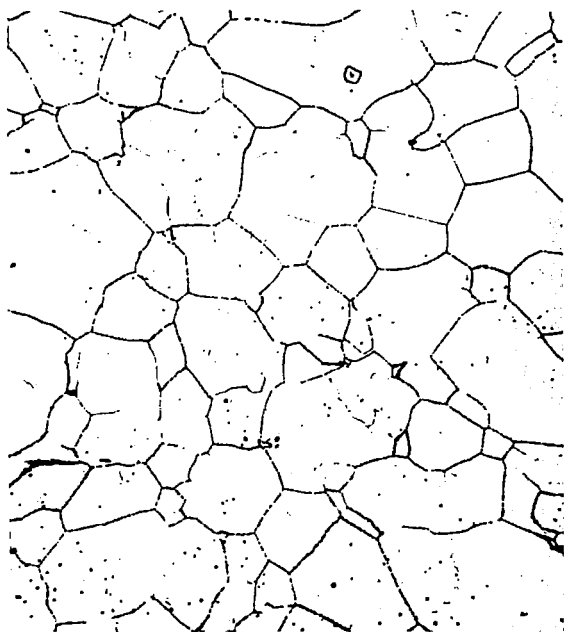
Solution Treated 1/2-hour at 1900°F



(d) 500X

Aged 10 hours at 1700°F

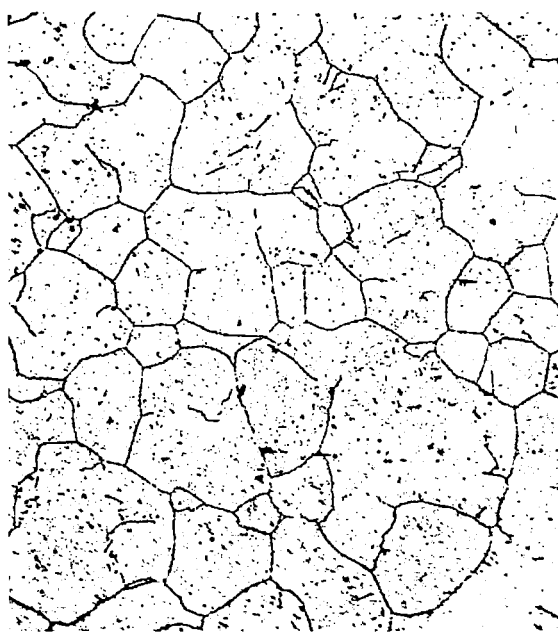
Figure 35. Optical photomicrographs of 0.026-inch thick Waspaloy sheet in various heat treated conditions.



(e) 500X

Aged 16 hours at 1400°F

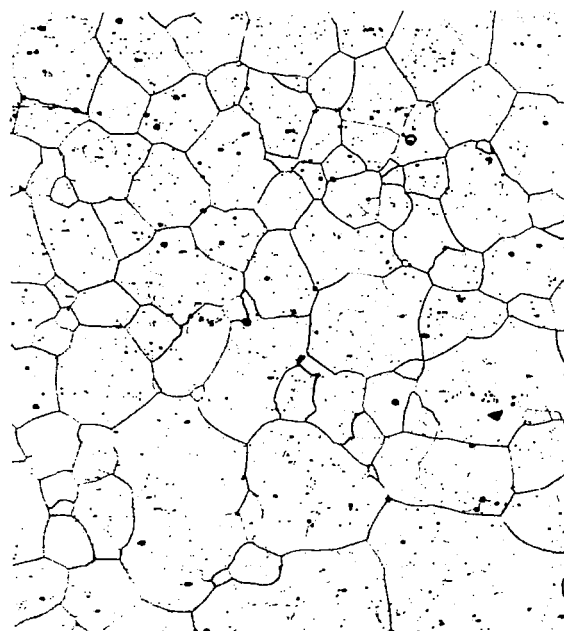
Solution Treated 1/2-hour at 1975°F



(f) 500X

Aged 10 hours at 1700°F

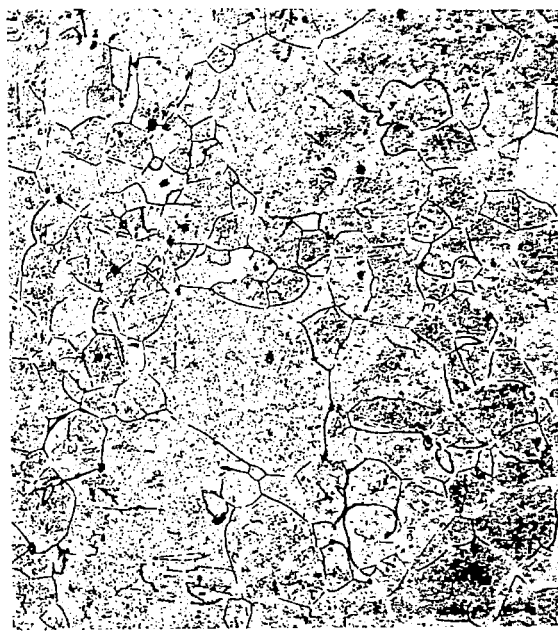
Solution Treated 1/2-hour at 1975°F



(g) 100X

Aged 16 hours at 1400°F

Solution Treated 1/2-hour at 2150°F



(h) 100X

Aged 10 hours at 1700°F

Solution Treated 1/2-hour at 2150°F

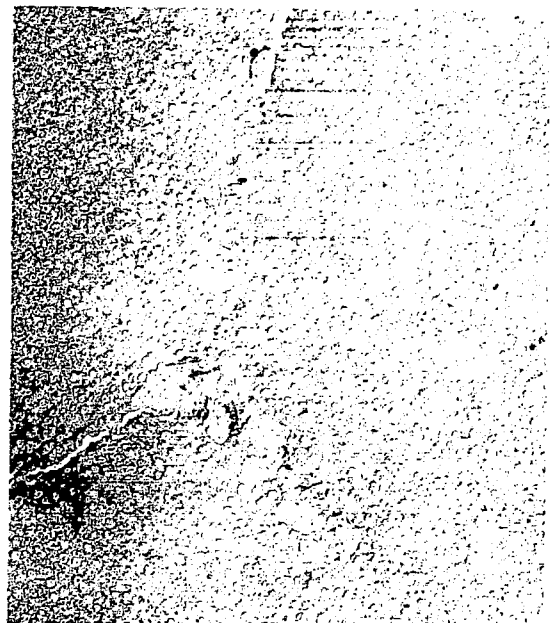
Figure 35 (Continued)



(a) 5,400X

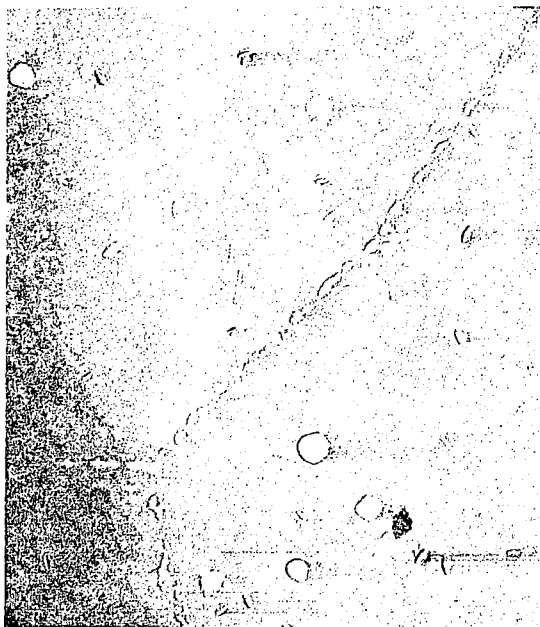
Aged 16 hours at 1400°F

Solution Treated 1/2-hour at 1825°F



(b) 5,400X

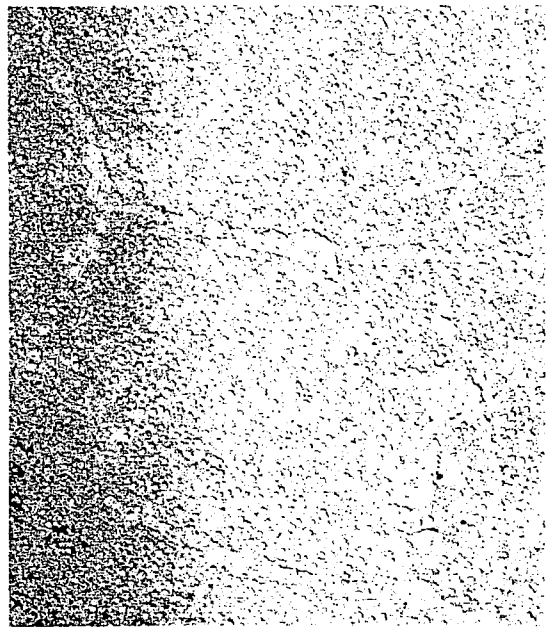
Aged 10 hours at 1700°F



(c) 5,400X

Aged 16 hours at 1400°F

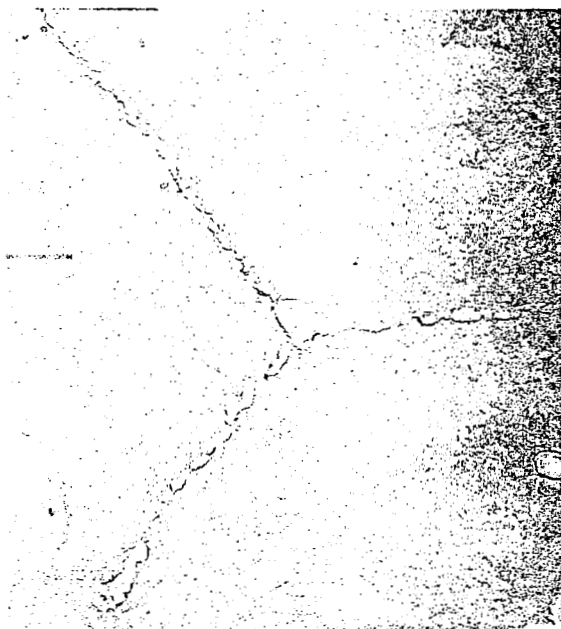
Solution Treated 1/2-hour at 1900°F



(d) 5,400X

Aged 10 hours at 1700°F

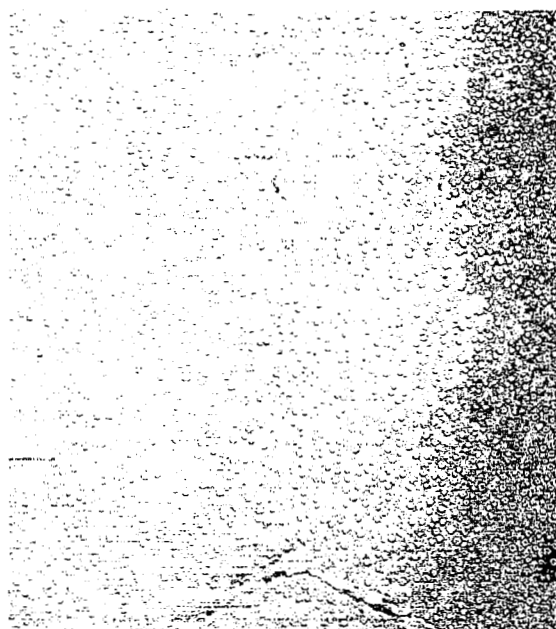
Figure 36. Replica electron micrographs of 0.026-inch thick Waspaloy sheet in the as-heat-treated condition.



(e) 5,400X

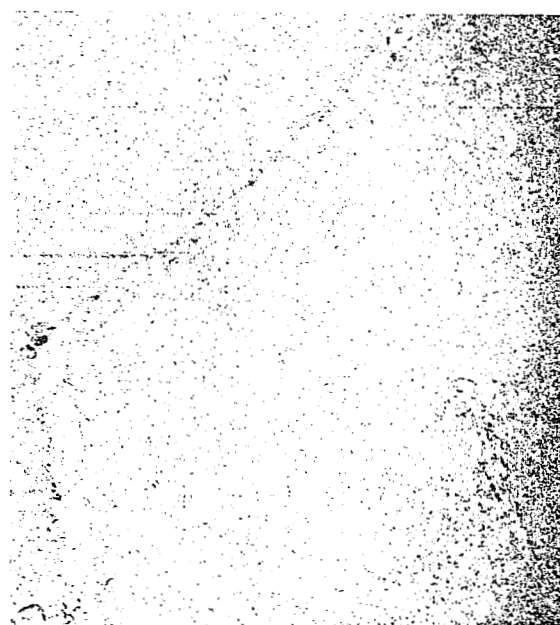
Aged 16 hours at 1400°F

Solution Treated 1/2-hour at 1975°F



(f) 5,400X

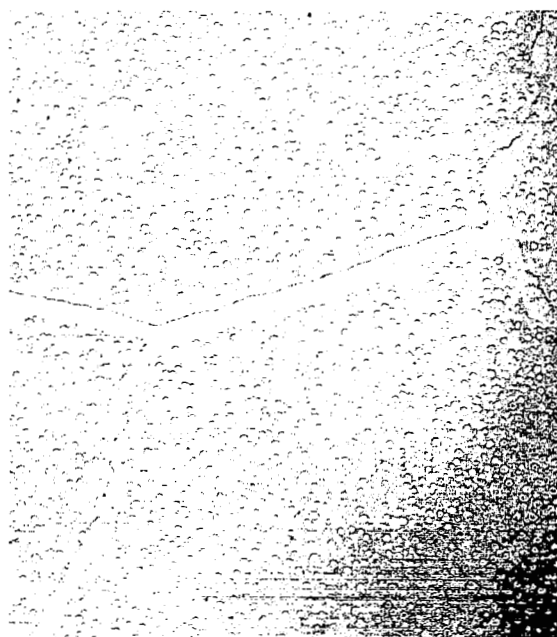
Aged 10 hours at 1700°F



(g) 5,400X

Aged 16 hours at 1400°F

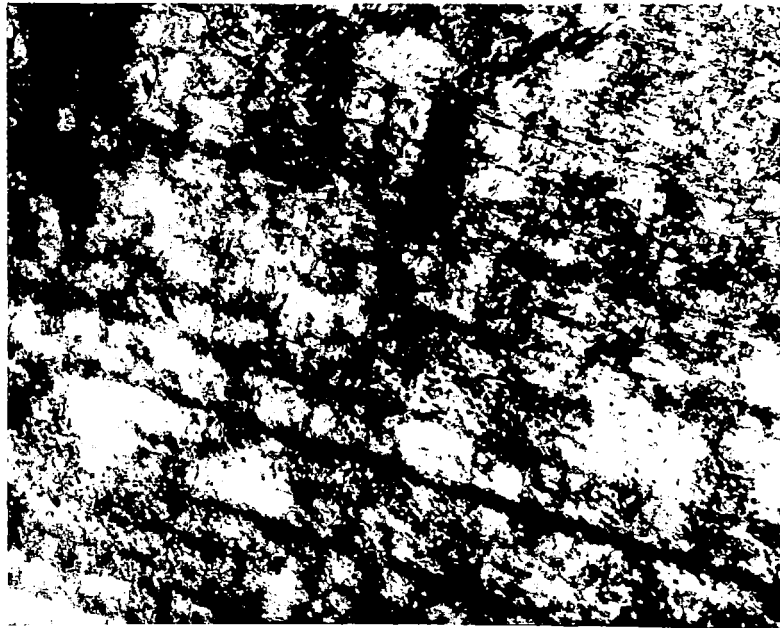
Solution Treated 1/2-hour at 2150°F



(h) 5,400X

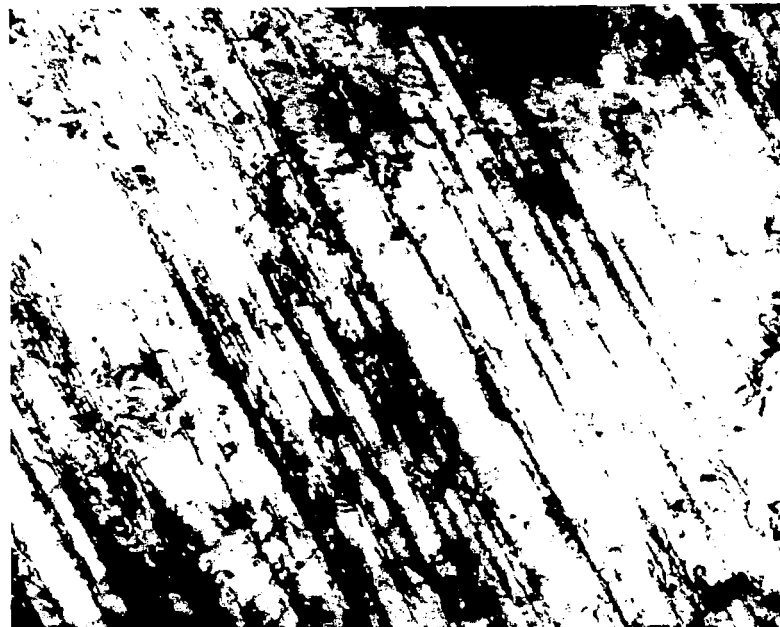
Aged 10 hours at 1700°F

Figure 36 (Continued)



(a)

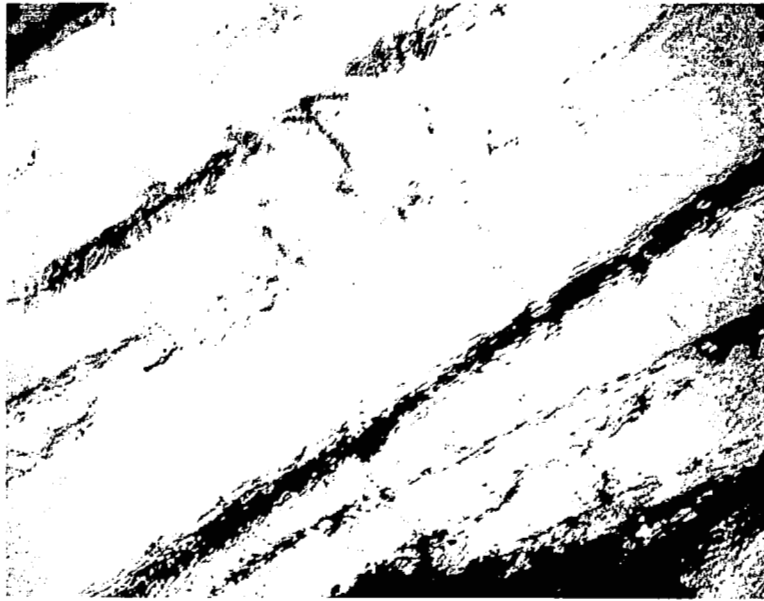
X24, 000



(b)

X49, 000

Figure 37. Thin-foil electron micrographs of Waspaloy, heat treated 1/2 hour at 1975°F, aged 16 hours at 1400°F and tensile tested at 1000°F (YS=115 ksi, TS=161 ksi, Elong. = 30%). Bands are evident that resulted from localized deformation.



(a)

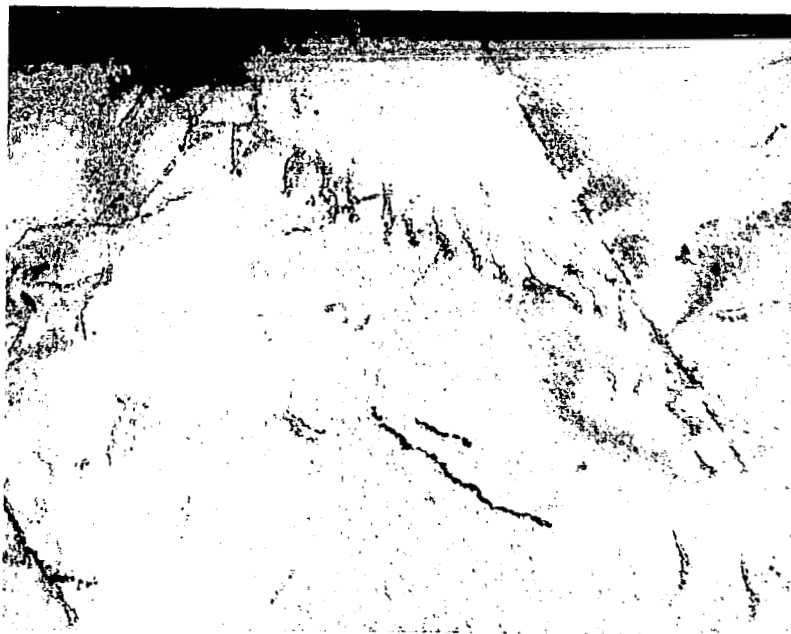
X27,000



(b)

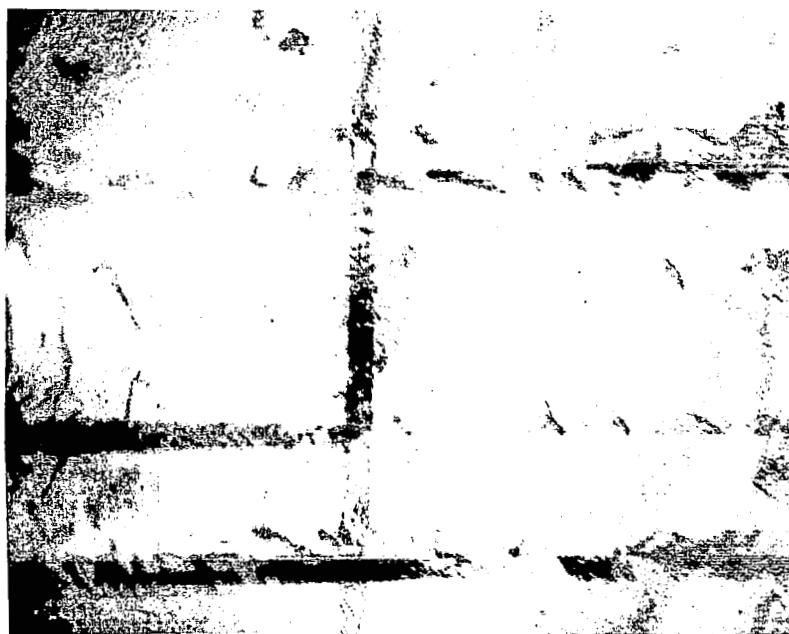
X22,000

Figure 38. Thin-foil electron micrographs of Waspaloy, heat treated 1/2 hour at 1975°F, aged 16 hours at 1400°F and creep-rupture tested at 135 ksi at 1000°F (ruptured in 517 hours at 7.0% elongation). The structures show deformation bands. Stacking faults, associated with extended dislocations, are also evident.



(a)

X42,000



(b)

X22,000

Figure 39. Transmission electron micrographs of Waspaloy, heat treated 1/2 hour at 1975°F, aged 16 hours at 1400°F and strained 2.3% at 1000°F. Dislocations are present in pile ups and are frequently "paired" to form superdislocations.



(a)

X11,000



(b)

X13,000

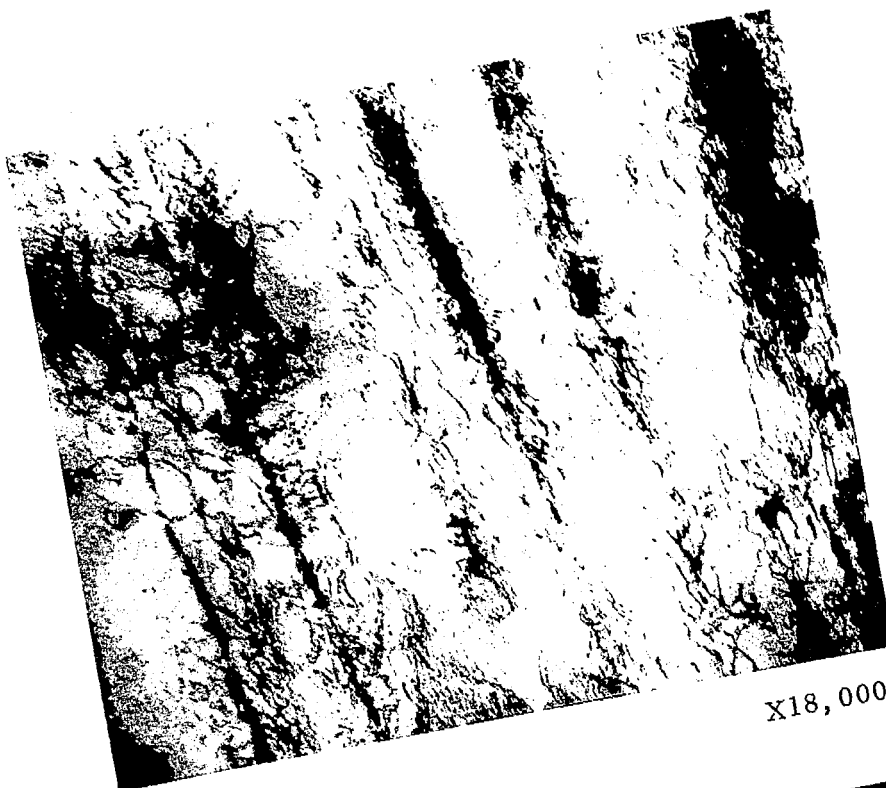


(c)

X19,000

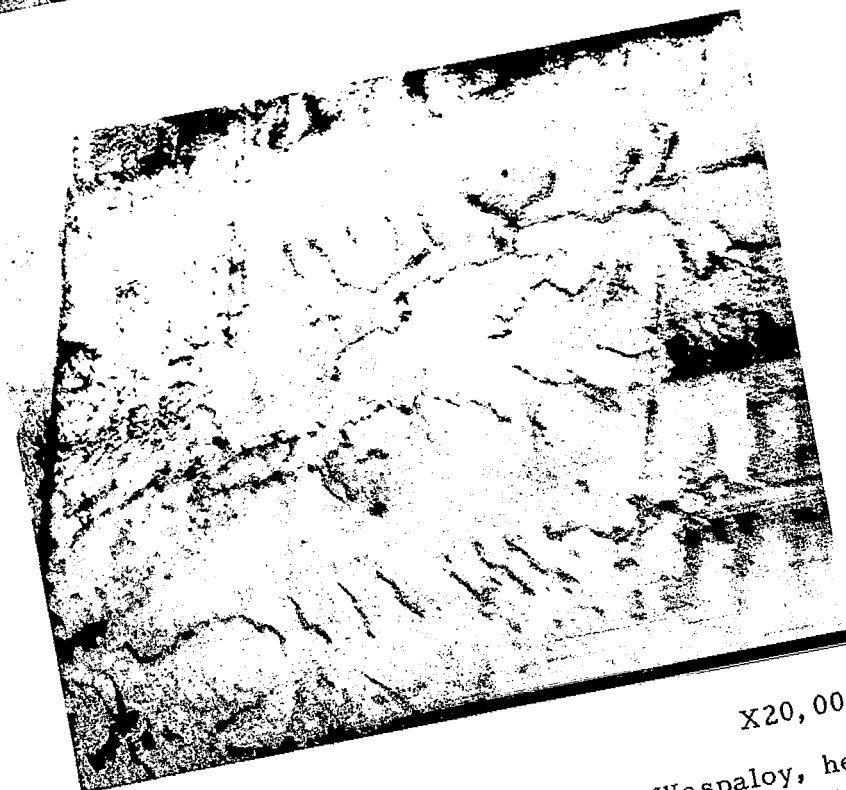
Figure 40. Thin-foil electron micrographs of Waspaloy, heat treated 1/2 hour at 1975°F, aged 16 hours at 1400°F and creep-rupture tested at 1200° and 1300°F. Dislocations are present in pile ups and are generally extended to form stacking fault ribbons. (a, b, - Tested at 90 ksi and 1200°F, ruptured in 376 hours at 1.5% elongation; c - Tested at 80 ksi at 1300°F, ruptured in 64 hours at 1.7% elongation.)





X18,000

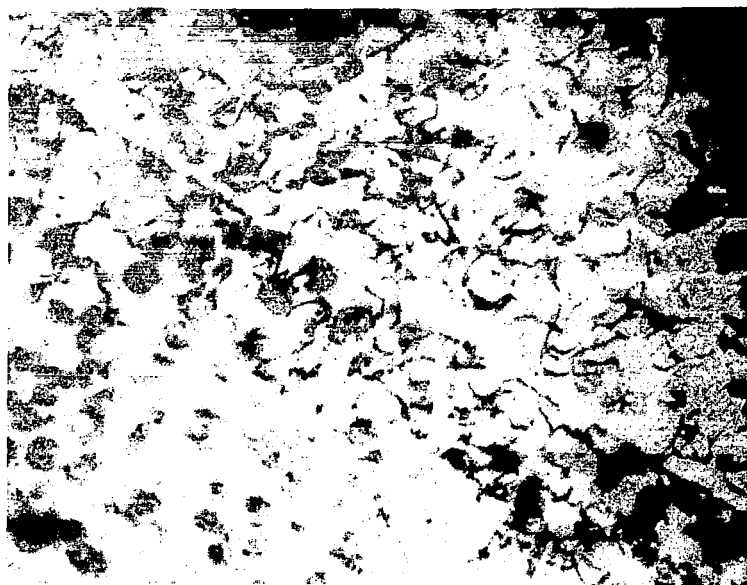
(a)



X20,000

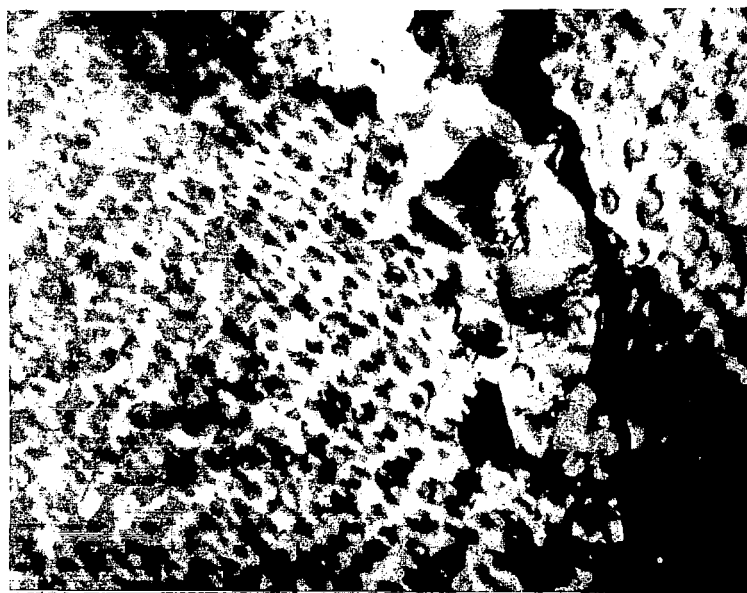
(b)

Thin-foil electron micrographs of Waspaloy, heat treated 1/1975°F, aged 16 hours at 1400°F and tensile tested 122 ksi, TS = 125 ksi, Elongation = 6%.



(a)

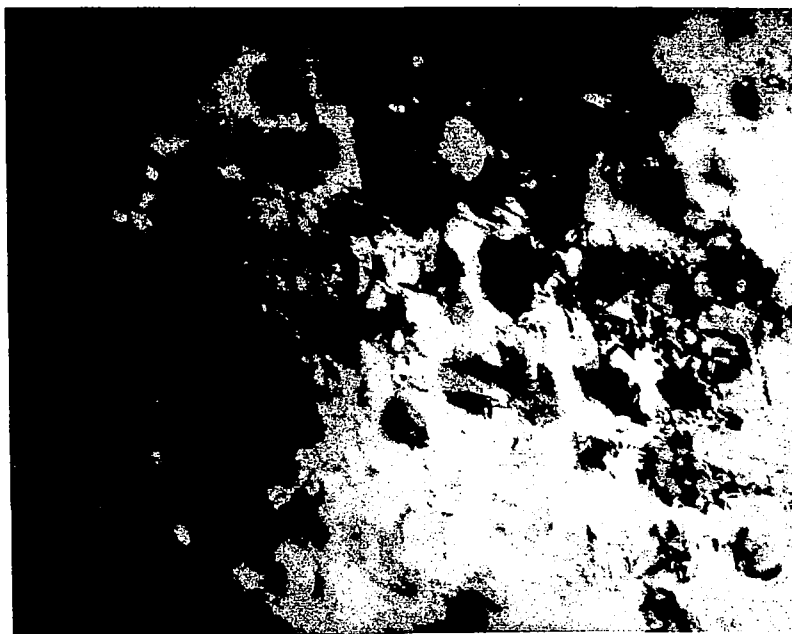
X67,000



(b)

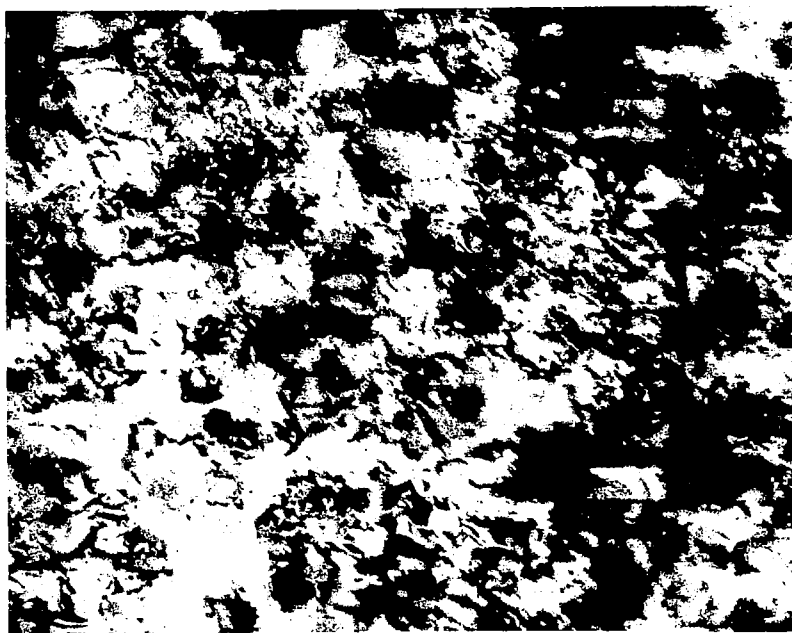
X56,000

Figure 42. Transmission electron micrographs of Waspaloy, heat treated 1/2 hour at 1975°F, aged 16 hours at 1400°F and creep-rupture tested at 38 ksi at 1400°F (ruptured in 1007 hours at 8.1% elongation). The  $\gamma'$  particles increased in size from approximately 75Å to 900Å during the test exposure. The deformation evident in (a) is homogeneous. Contrast effects associated with coherent  $\gamma'$  are present in (b).



(a)

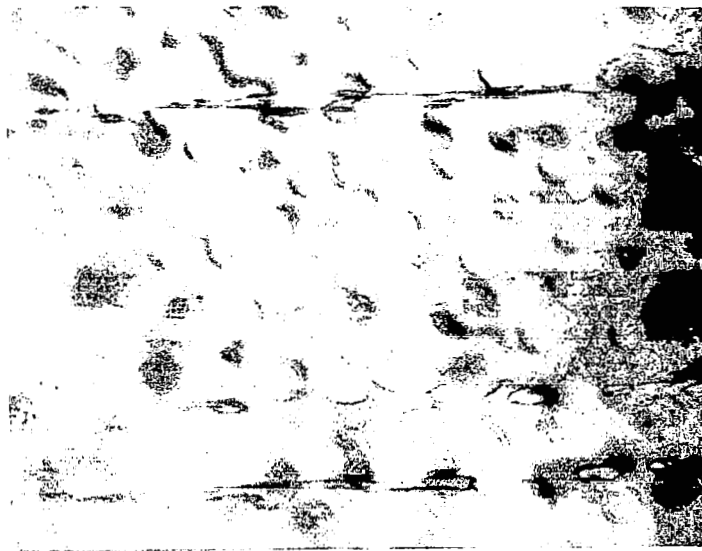
75,000X



(b)

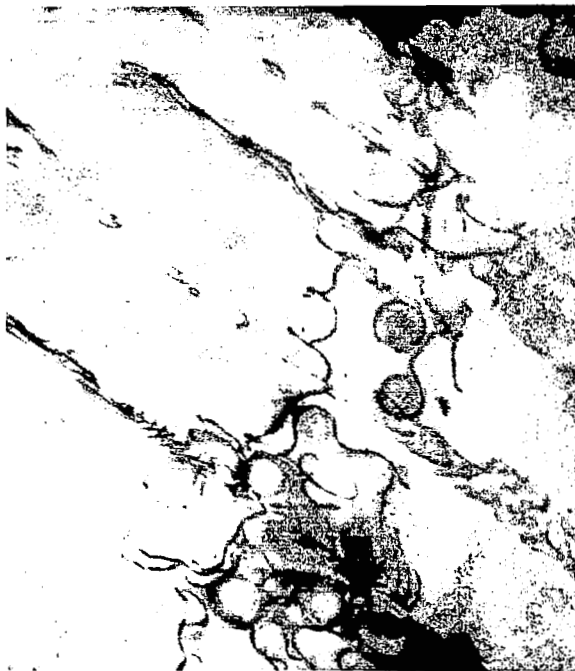
51,000X

Figure 43. Transmission electron micrographs of Waspaloy, heat treated 1/2 hour at 1975°F, aged 10 hours at 1700°F and tensile tested at 1000°F (YS = 95 ksi, TS = 148 ksi, Elongation = 32%). The structures are highly deformed. There was a tendency for the deformation to be maximized in slip bands.



(a)

X51,000



(b)

X47,000



(c)

X38,000

Figure 44. Thin-foil electron micrographs of Waspaloy, heat treated 1/2 hour at 1975°F, aged 10 hours at 1700°F and strained approximately 1, 2, 4, and 6% at 1000°F (a, b, c respectively). Dislocations can be observed bowing between  $\gamma'$  particles leaving "pinched off" dislocation loops around the particles. At the high strain level the deformation, although generally "homogeneous", does tend to be concentrated in slip bands.



(a)

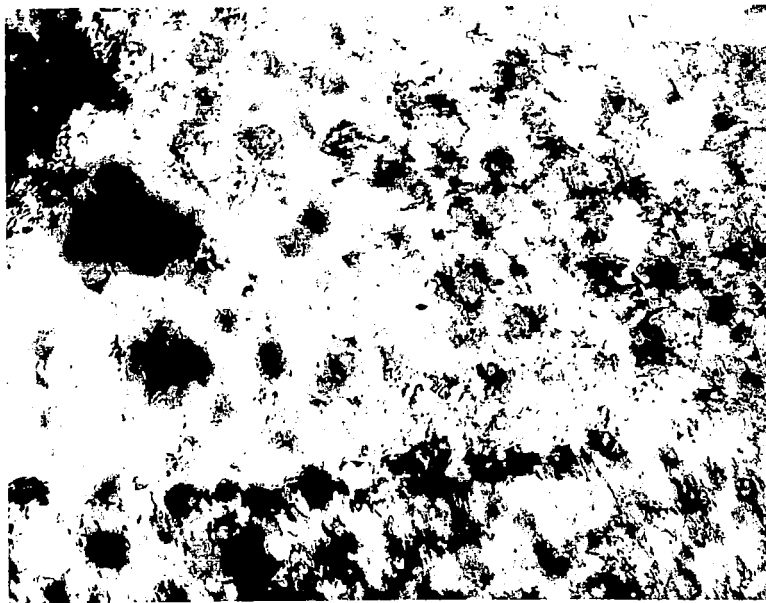
X55,000



(b)

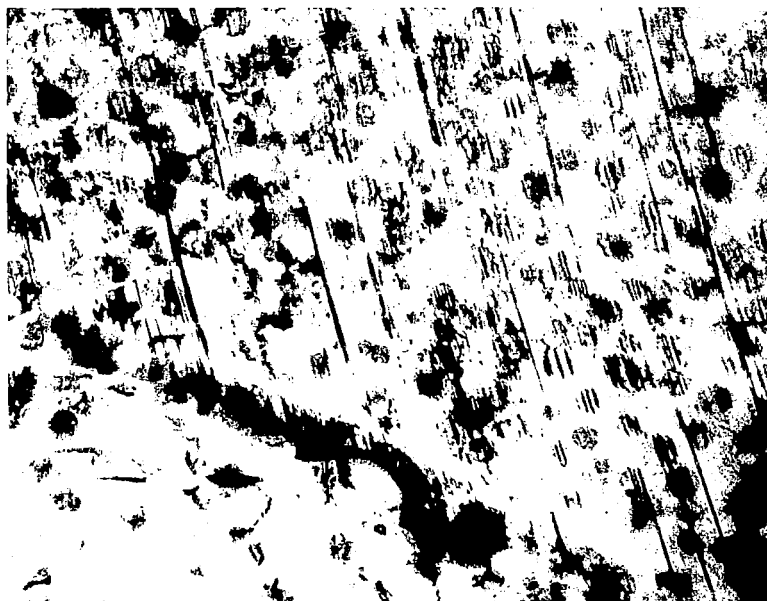
X50,000

Figure 45. Thin-foil electron micrographs of Waspaloy, heat treated 1/2 hour at 1975°F, aged 10 hours at 1700°F and creep-rupture tested at 1000° and 1200°F. The deformation is "homogeneous". Microtwins are present in (a) while dislocation loops about  $\gamma'$  particles are evident in (b). (a - tested at 125 ksi at 1000°F, ruptured in 566 hours at 14.3% elongation; b - tested at 80 ksi at 1200°F, ruptured in 1870 hours at 5.4% elongation.)



(a)

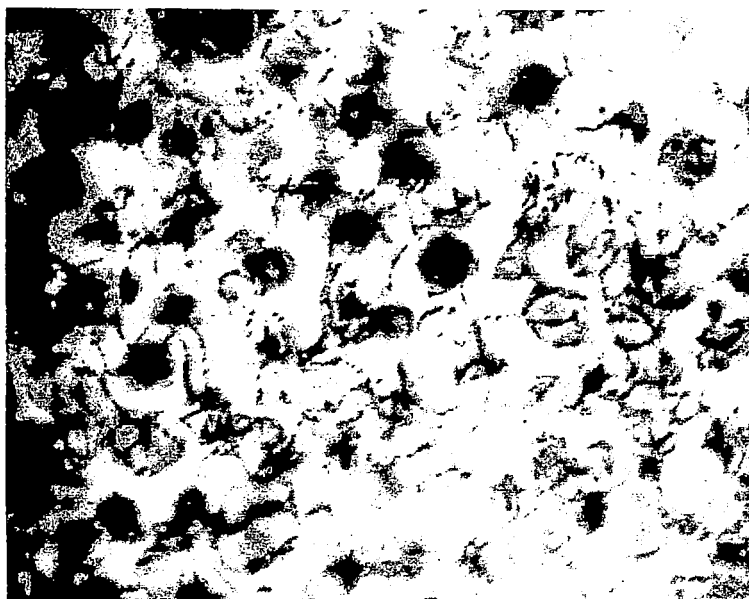
X43,000



(b)

X38,000

Figure 46. Thin-foil electron micrographs of Waspaloy, heat treated 1/2 hour at 1975°F, aged 10 hours at 1700°F and tensile tested at 1400°F (YS = 92 ksi, TS = 115 ksi, Elongation = 15%). The dislocations are "homogeneously" distributed - (a). In areas of (b) microtwins in the  $\gamma'$  and matrix  $\gamma$  are apparent.



(a)

X33,000



(b)

X36,000

Figure 47. Transmission electron micrographs of Waspaloy, heat treated 1/2 hour at 1975°F, aged 10 hours at 1700°F and creep-rupture tested at 38 ksi at 1400°F (ruptured in 931 hours at 9% elongation). The  $\gamma'$  particles increased in size from about 1000Å to 1500Å during the test exposure. A "homogeneous" distribution of dislocations is evident in (a), while in (b) contrast effects due to coherent  $\gamma'$  particles are discernible.

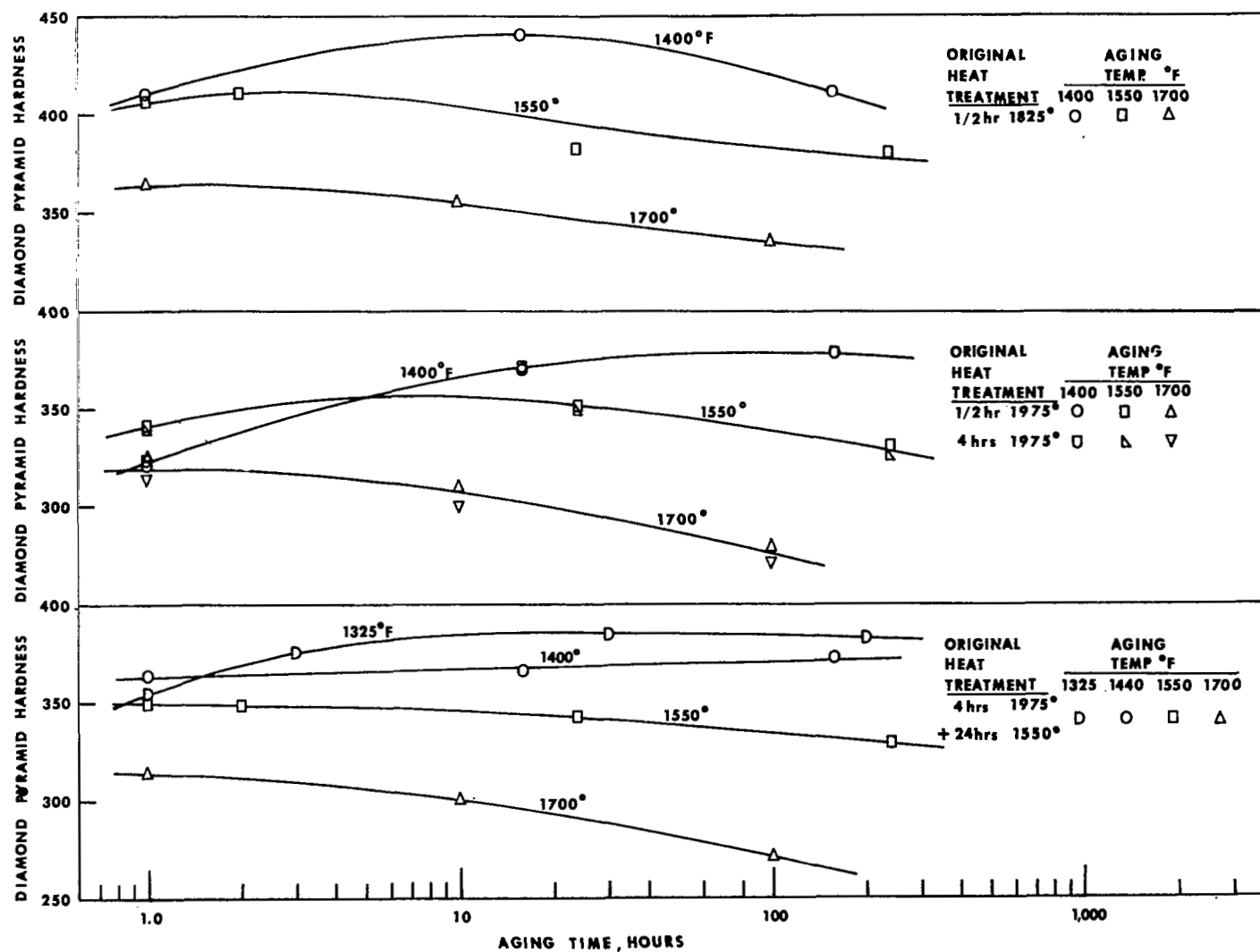
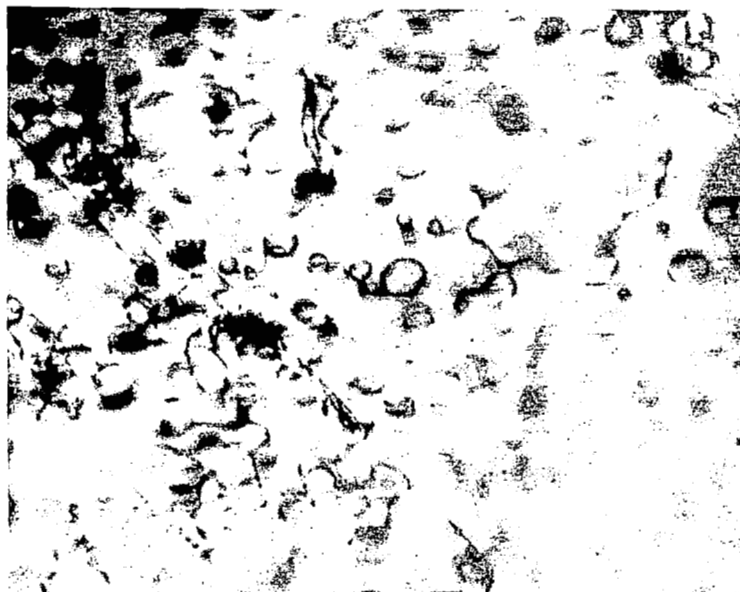


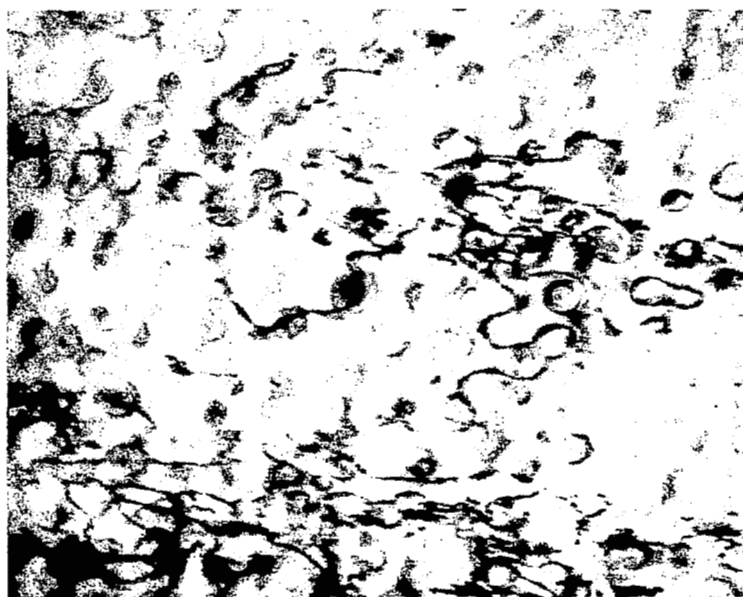
Figure 48. Effect of aging exposures at 1325°, 1400°, 1550° and 1700°F on the Diamond Pyramid Hardness of several selected heat treatments of 0.026-inch thick Waspaloy sheet.





46,000X

Figure 49. Thin-foil electron micrographs of Waspaloy heat treated 1/2 hour at 1975°F and aged 24 hours at 1550°F and strained about 2% at 1000°F. Bowing of dislocations between the  $\gamma'$  particles resulted in the "pinched off" dislocation loops evident.



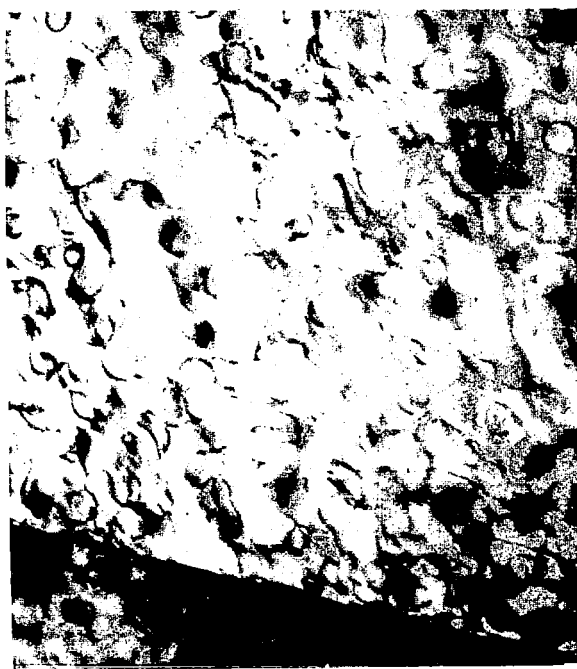
52,000X

Figure 50. Transmission electron micrographs of Waspaloy heat treated 4 hours at 1975°F and aged 24 hours at 1550° plus 16 hours at 1400°F and strained approximately 2% at 1000°F. The dislocation structures are similar to those of Figure 49.



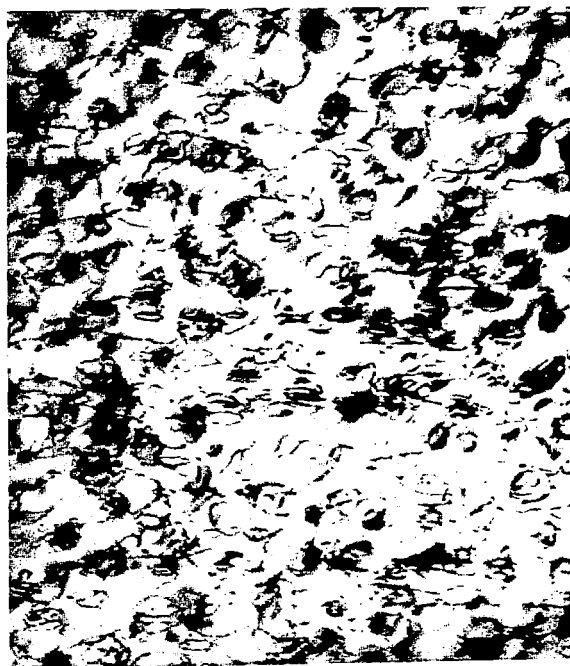
(a)

63,000X



(b)

51,000X



(c)

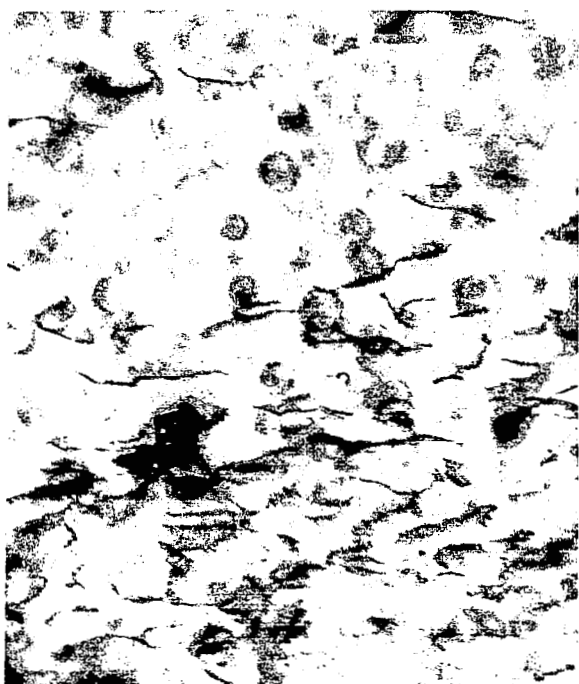
40,000X

Figure 51. Thin-foil electron micrographs of Waspaloy heat treated 1/2 hour at 1975°F and aged 24 hours at 1550° and creep-rupture tested at 80 ksi at 1200°F (ruptured in 2483 hours at 4.5 percent elongation) a - Contrast effects due to coherency of the  $\gamma'$ . b, c - Homogeneous distributions of dislocations bowing between  $\gamma'$  particles leaving "pinched off" dislocation loops.



(a)

41,000X



(b)

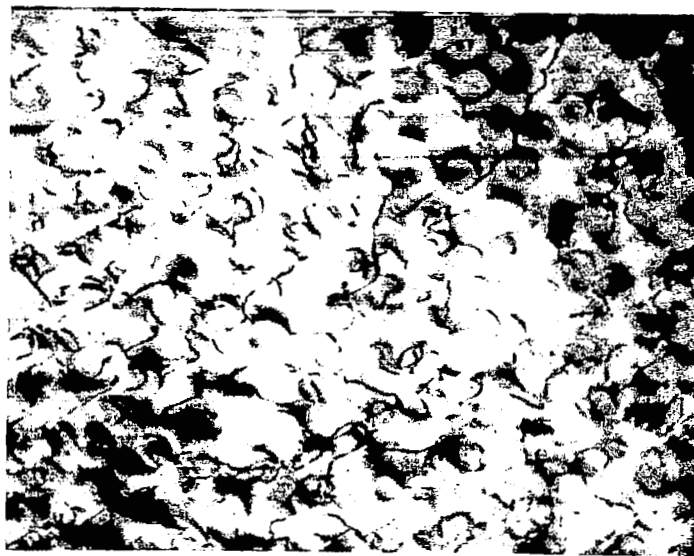
49,000X



(c)

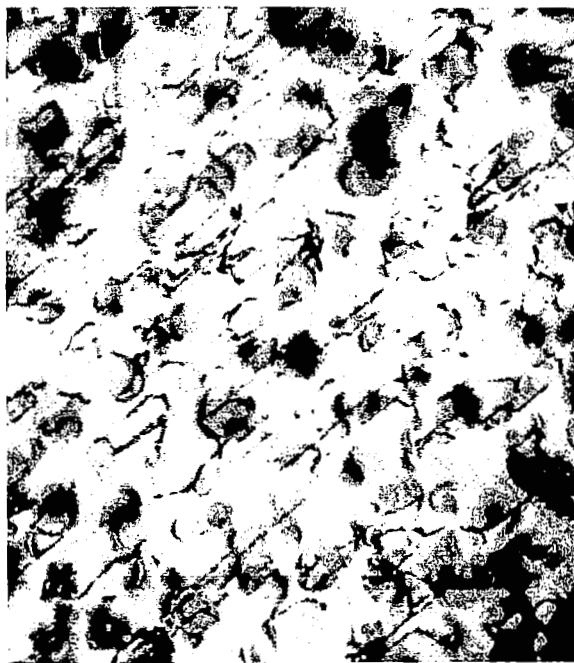
24,000X

Figure 52. Thin-foil electron micrographs of Waspaloy heat treated 4 hours at 1975°F and aged 24 hours at 1550°F plus 16 hours at 1400°F and creep-rupture tested at 80 ksi at 1200°F (ruptured in 1706 hours at 2.6 percent elongation). The dislocation structures are similar to those of Figure 51.



(a)

45,000X



(b)

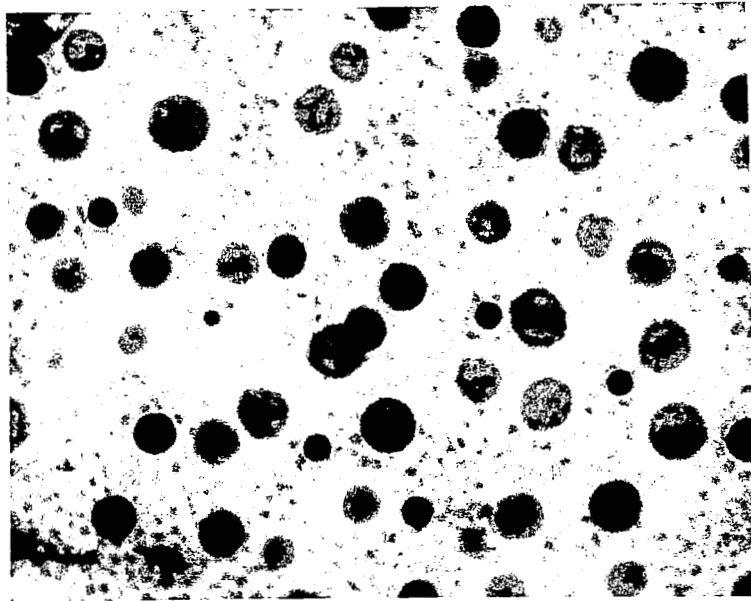
53,000X



(c)

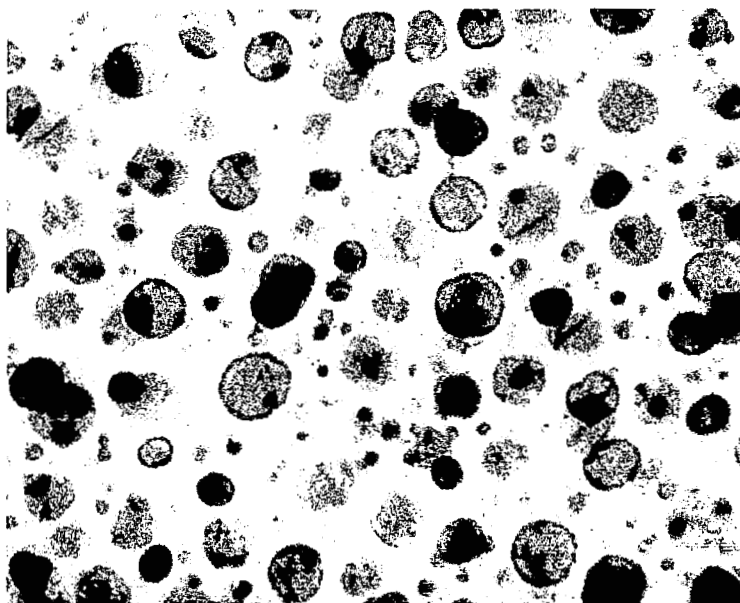
60,000X

Figure 53. Transmission electron micrographs of Waspaloy heat treated 1/2 hour at 1975°F and aged 24 hours at 1550°F and creep-rupture tested at 38 ksi at 1400°F (ruptured in 872 hours at 9.3 percent deformation). The following principal features are evident: a, b - Homogeneous dislocating distribution. c - Contrast effect associated with the coherent  $\gamma'$  particles.



(a)

43,000X



(b)

32,000X

Figure 54. Transmission electron micrographs showing  $\gamma'$  size distributions of Waspaloy heat treated 1/2 hour at 1825°F and aged 16 hours at 1400°F and creep-rupture tested at 1100° and 1300°F (a - Tested at 120 ksi and 1100°F, ruptured in 3510 hours. b - Tested at 60 ksi and 1300°F, ruptured in 1239 hours). The two distinctly different  $\gamma'$  sizes present in the as-heat treated material tended to merge with increasing time and temperature of the test exposure, for example from (a) to (b).



(a)

43,000X



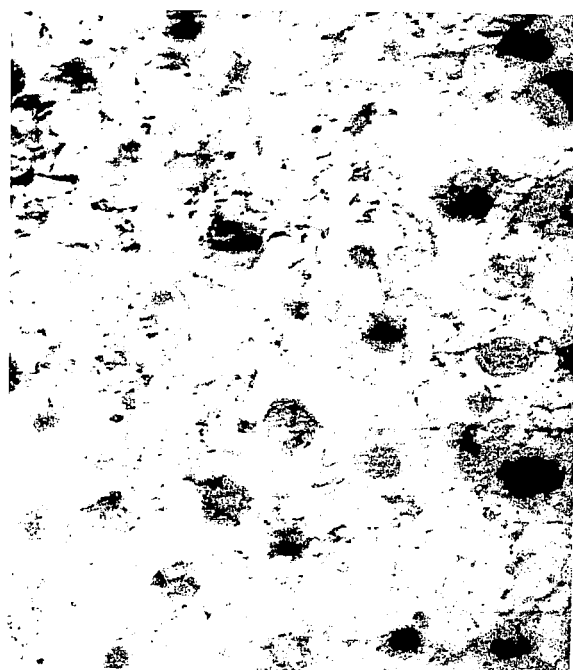
(b)

62,000X



(c)

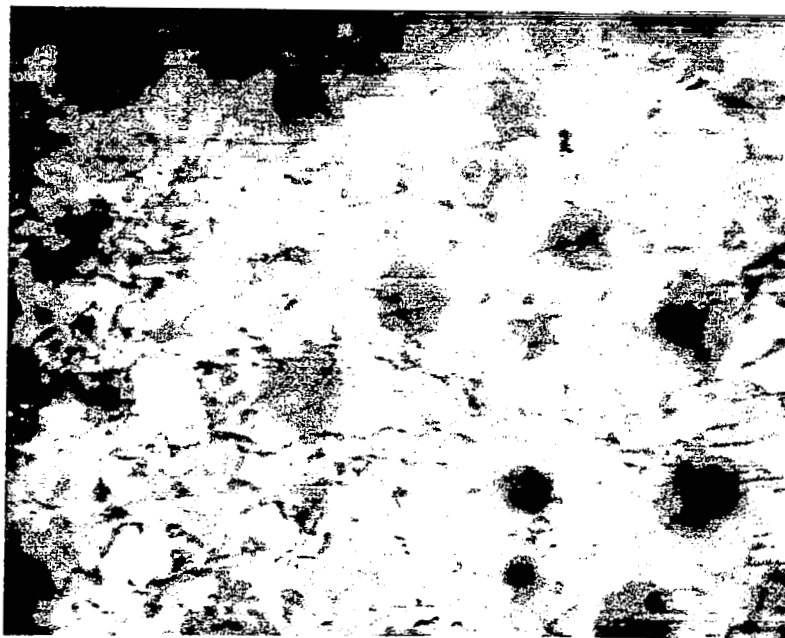
56,000X



(d)

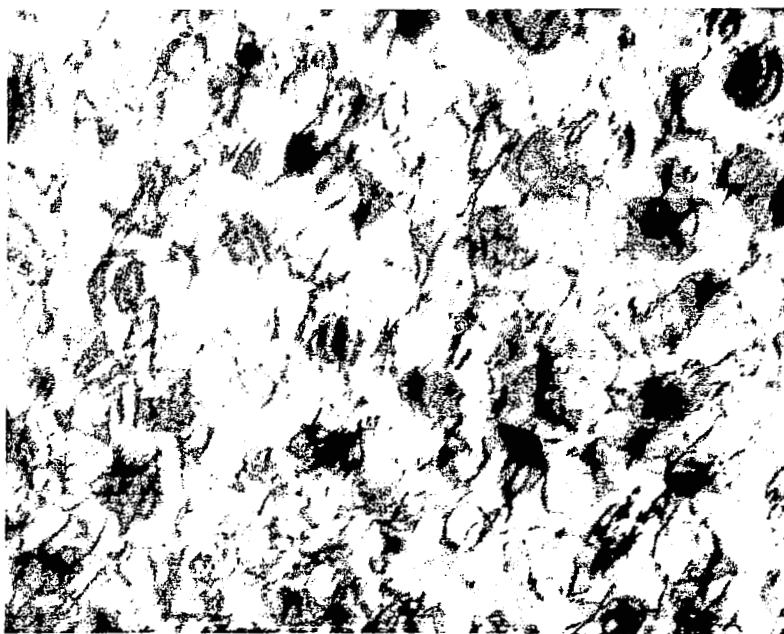
44,000X

Figure 55. Transmission electron micrographs of Waspaloy heat treated .1/2 hour at 1825°F and aged 16 hours at 1400°F, and creep-rupture tested at 1000° and 1100°F. Within the homogeneous deformation microtwins and stacking faults can be observed. (a, b, and c, - tested at 165 ksi at 1000°F ruptured in 480 hours. d - tested at 120 ksi at 1100°F ruptured in 3510 hours).



(a)

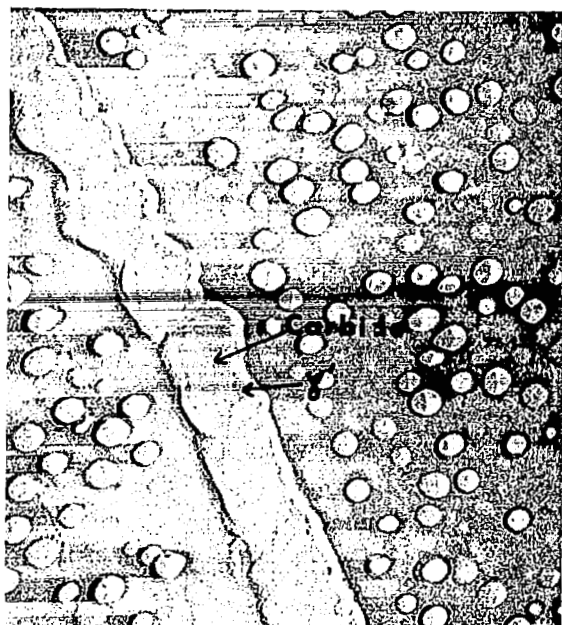
50,000X



(b)

43,000X

Figure 56. Thin-foil electron micrographs of Waspaloy heat treated 1/2 hour at 1825°F and aged 16 hours at 1400°F and creep-rupture tested at 1200° and 1300°F. The deformation resulted in an homogeneous distribution of dislocations. (a - Tested at 85 ksi at 1200°F, ruptured in 2560 hours at 3.4% elongation. b - Tested at 60 ksi and 1300°F, ruptured in 1239 hours at 6.2% elongation).

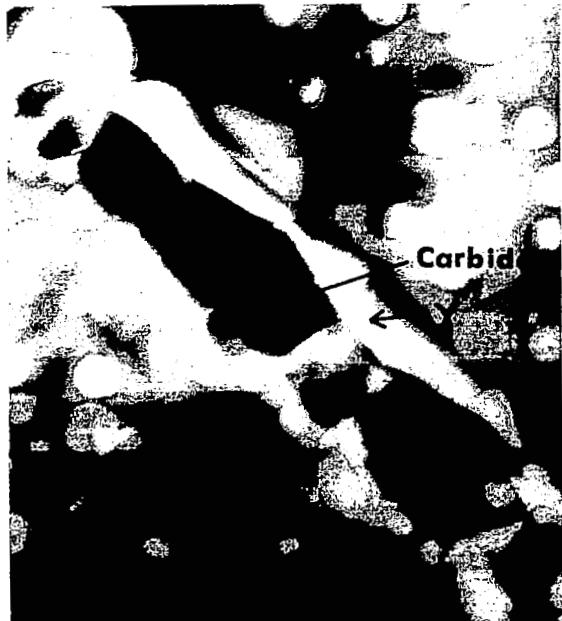


(a) 16,000X



(b) 14,000X

Figure 57. Electron micrographs showing  $\gamma'$  and carbide precipitates in the grain boundary of 0.026-inch thick Waspaloy sheet solution treated 1/2-hour at 2150°F and aged 10 hours at 1700°F.



(a) 38,000X



(b) 44,000X

Figure 58. Transmission electron micrographs showing  $\gamma'$  and carbide precipitates in the grain boundary of 0.026-inch thick Waspaloy sheet solution treated 1/2-hour at 2150°F and aged 10 hours at 1700°F.



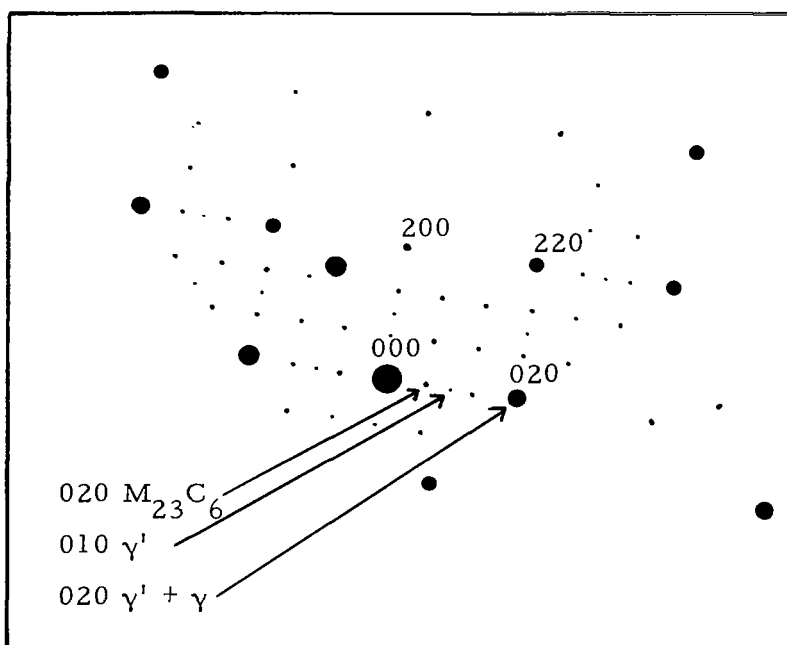
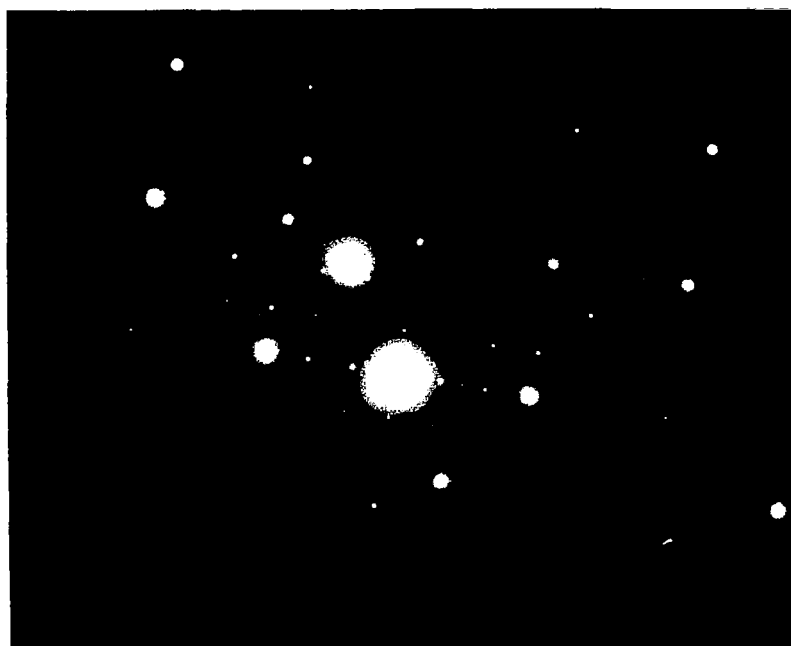
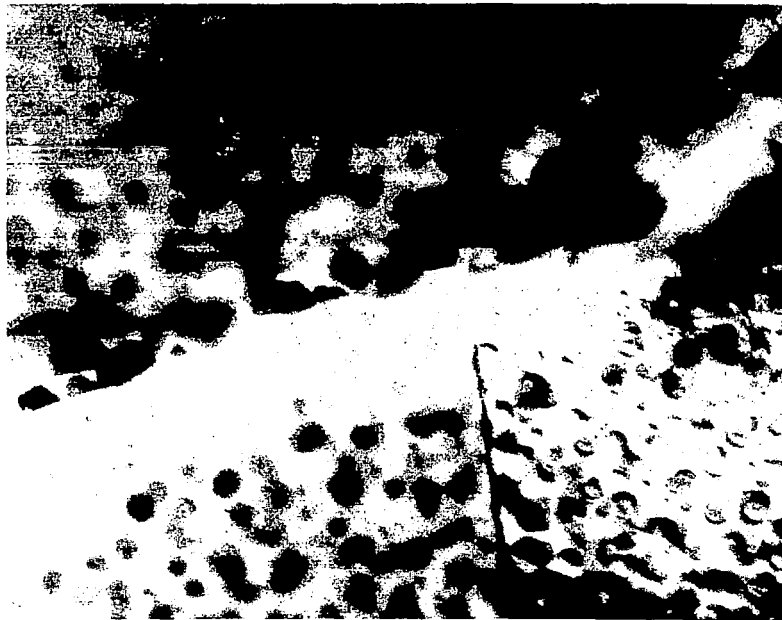


Figure 59. Selected area diffraction pattern of a grain boundary (0.026-inch thick Waspaloy sheet solution treated 1/2-hour at 2150°F and aged 16 hours at 1400°F) showing identical orientations of  $M_{23}C_6$  and the matrix on one side of the boundary.



(a)

X35,000



(b)

X27,000

Figure 60. Transmission electron micrographs of Waspaloy, heat treated 1/2 hour at 1975°F, aged, and creep-rupture tested at 38 ksi at 1400°F. The zones denude of the  $\gamma'$  precipitate formed during the creep exposure. (a- Aged 16 hours at 1400°F, ruptured in 1007 hours; b - Aged 10 hours at 1700°F, ruptured in 931 hours).

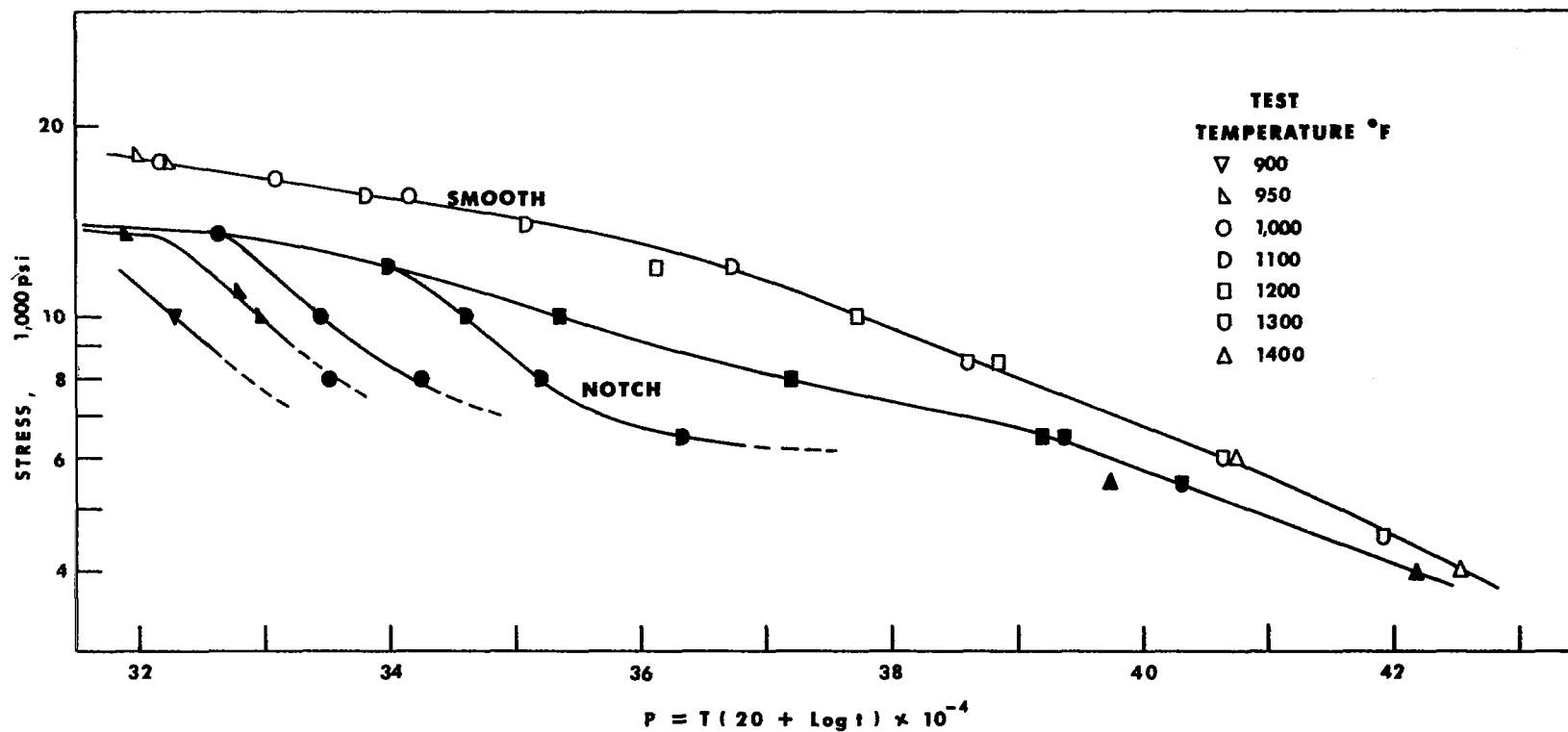


Figure 61. Time-temperature dependence of the rupture strengths at temperatures from 900° to 1400°F of smooth and notched specimens of 0.026-inch thick Waspaloy sheet heat treated 1/2 hour at 1825°F and aged 16 hours at 1400°F.

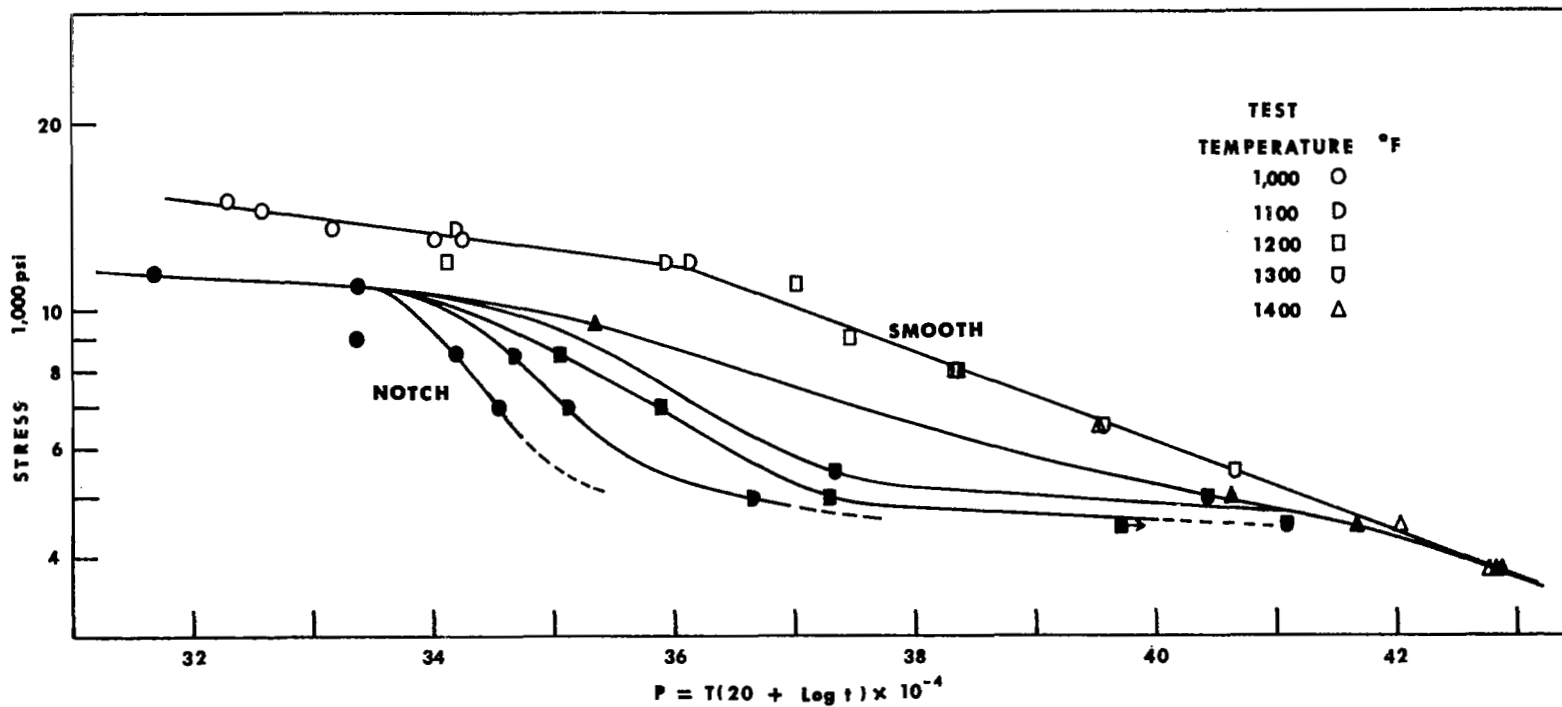


Figure 62. Time-temperature dependence of the rupture strengths at temperatures from 1000° to 1400°F of smooth and notched specimens of 0.026-inch thick Waspaloy sheet heat treated 1/2 hour at 1975°F and aged 16 hours at 1400°F.

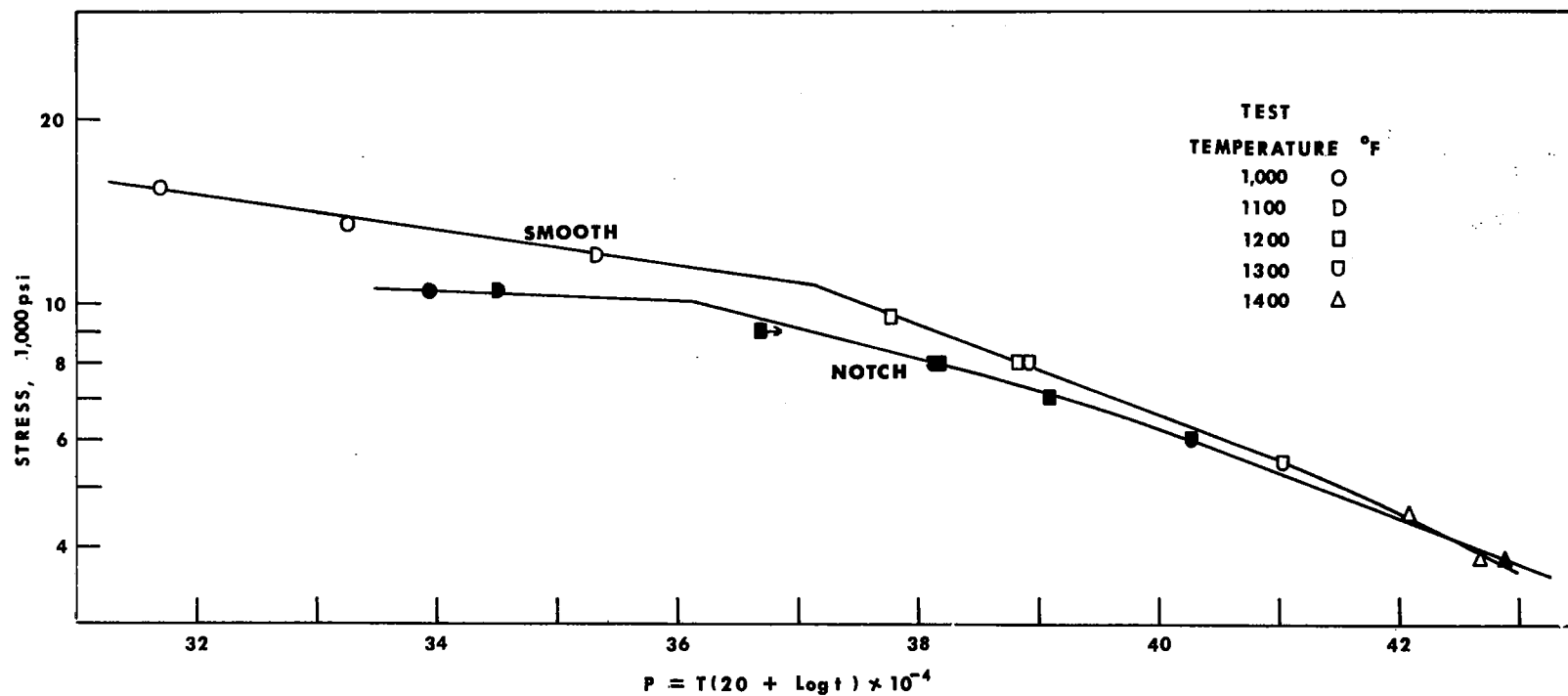


Figure 63. Time-temperature dependence of the rupture strengths at temperatures from 1000° to 1400°F of smooth and notched specimens of 0.026-inch thick Waspaloy sheet heat treated 1/2 hour at 1975°F and aged 24 hours at 1550°F.

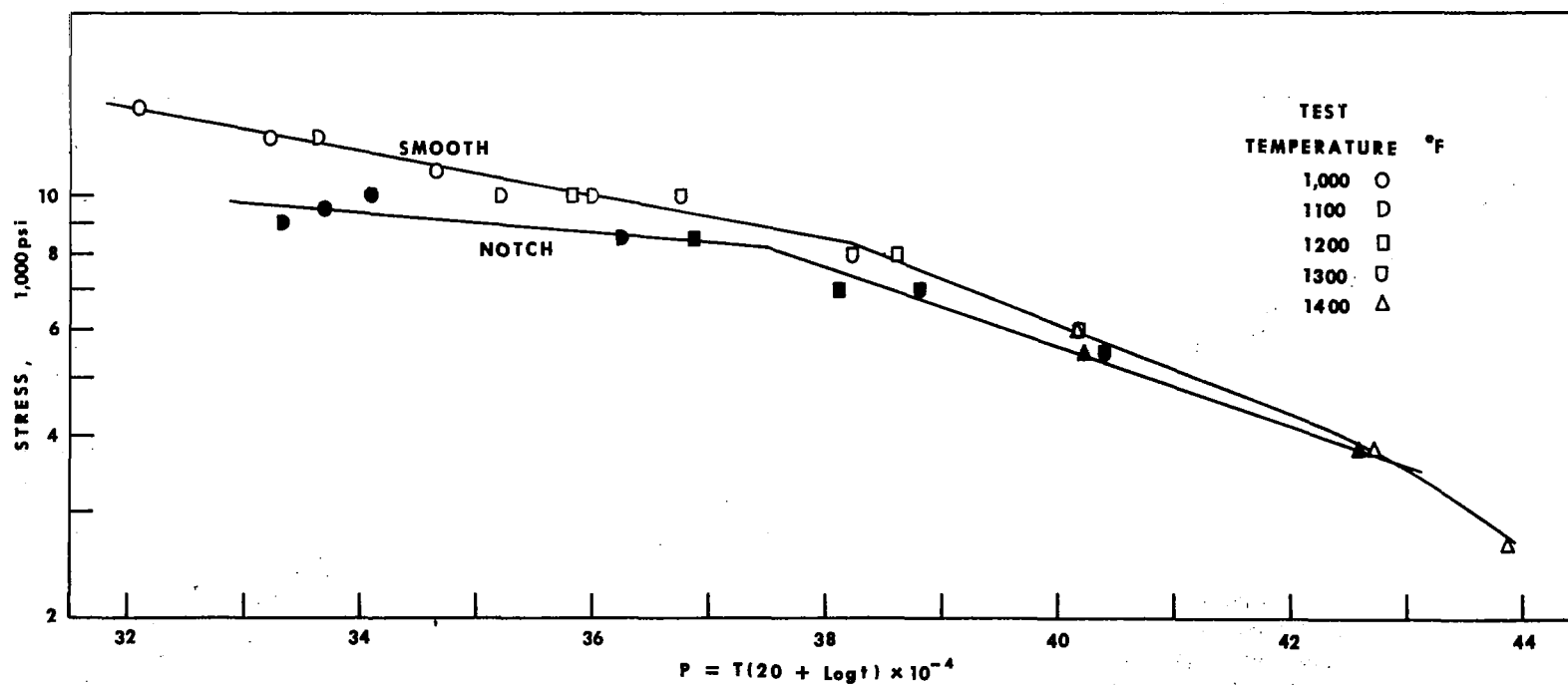


Figure 64. Time-temperature dependence of the rupture strengths at temperatures from 1000° to 1400°F of smooth and notched specimens of 0.026-inch thick Waspaloy sheet heat treated 1/2 hour at 1975°F and aged 10 hours at 1700°F.

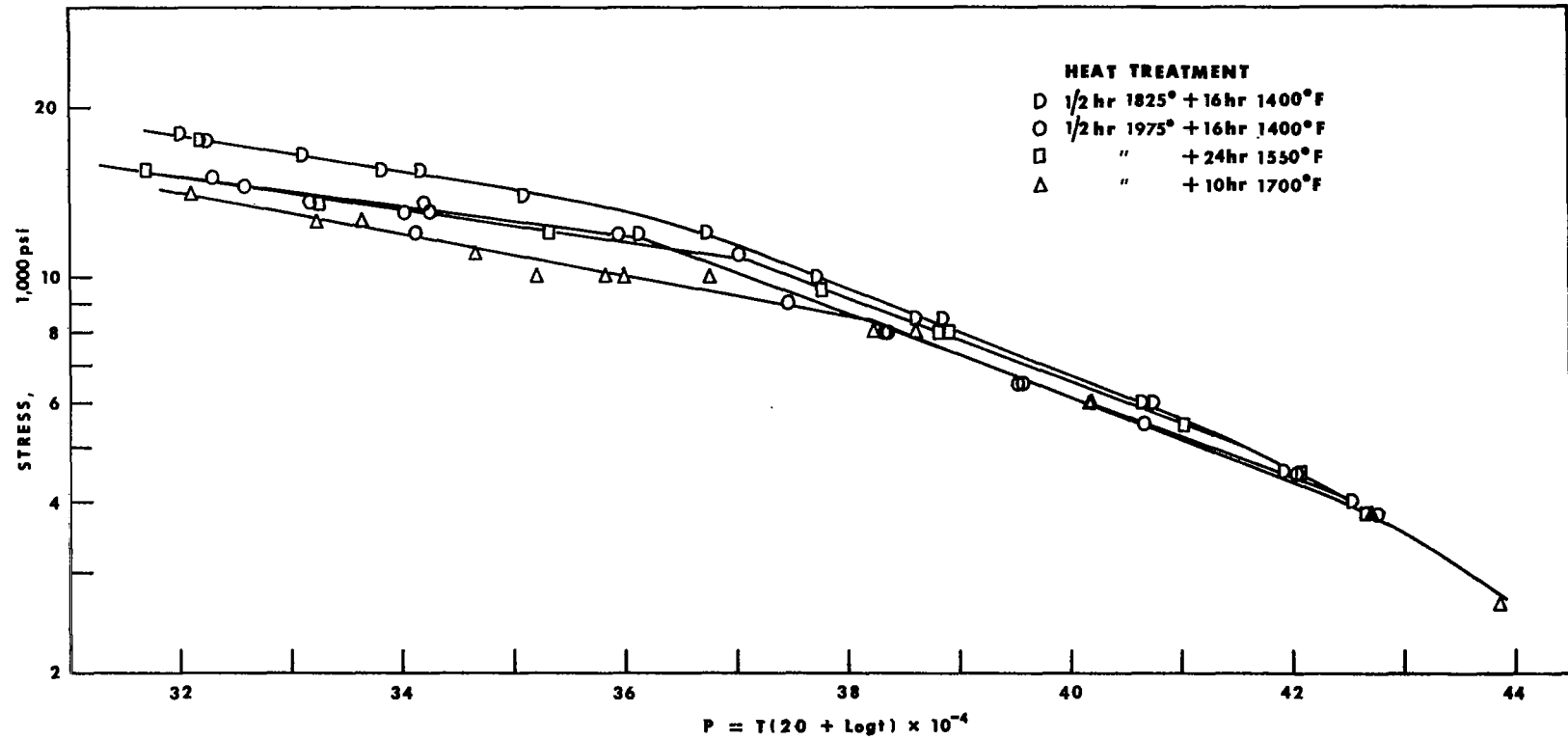


Figure 65. Time-temperature dependence of the rupture strengths at temperatures from 1000° to 1400°F of smooth specimens of 0.026-inch thick Waspaloy sheet in the heat treated conditions.

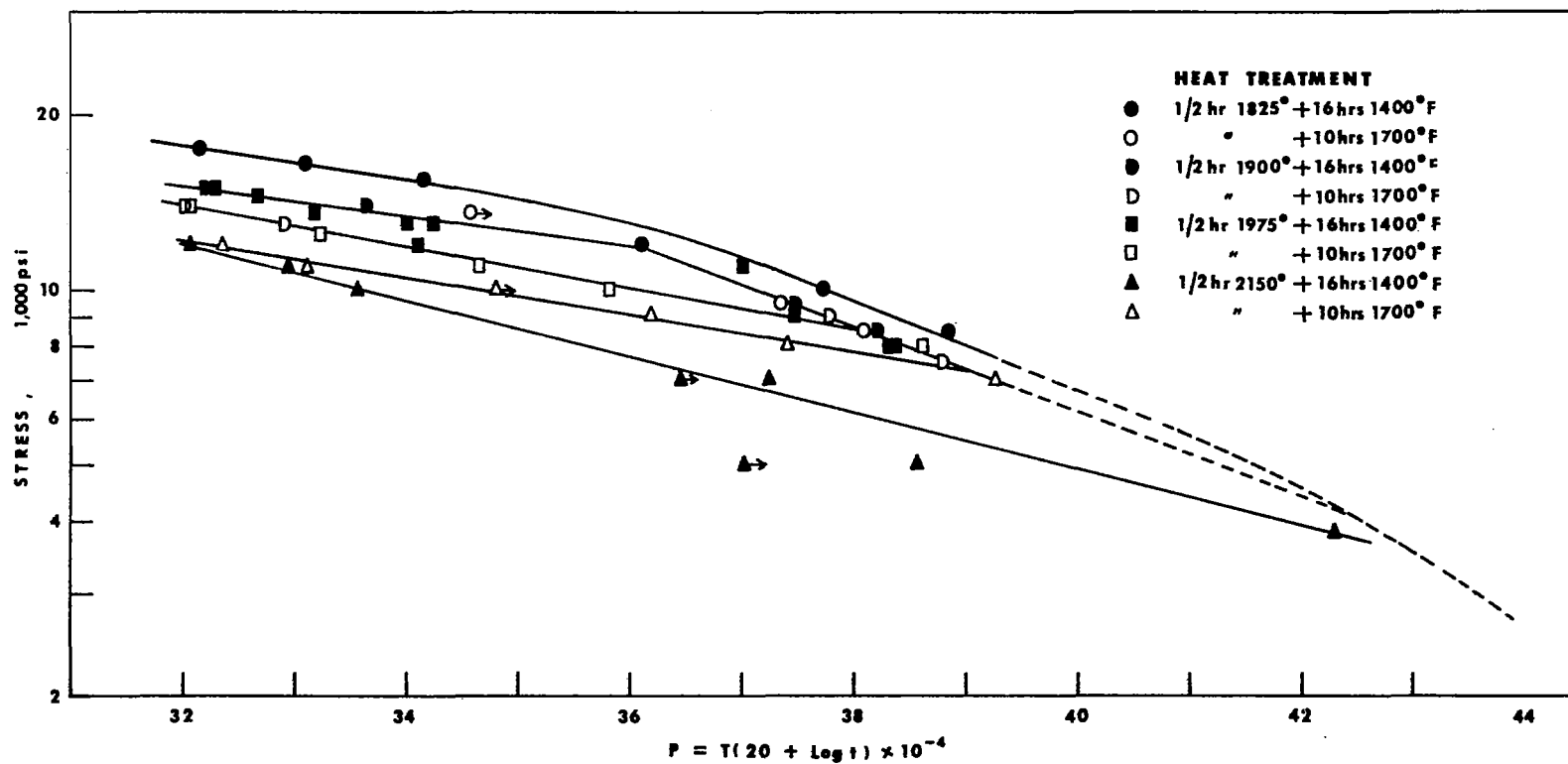


Figure 66. Time-temperature dependence of the rupture strengths at temperatures of 1000° and 1200°F of smooth specimens of 0.026-inch thick Waspaloy sheet in the heat treated conditions.



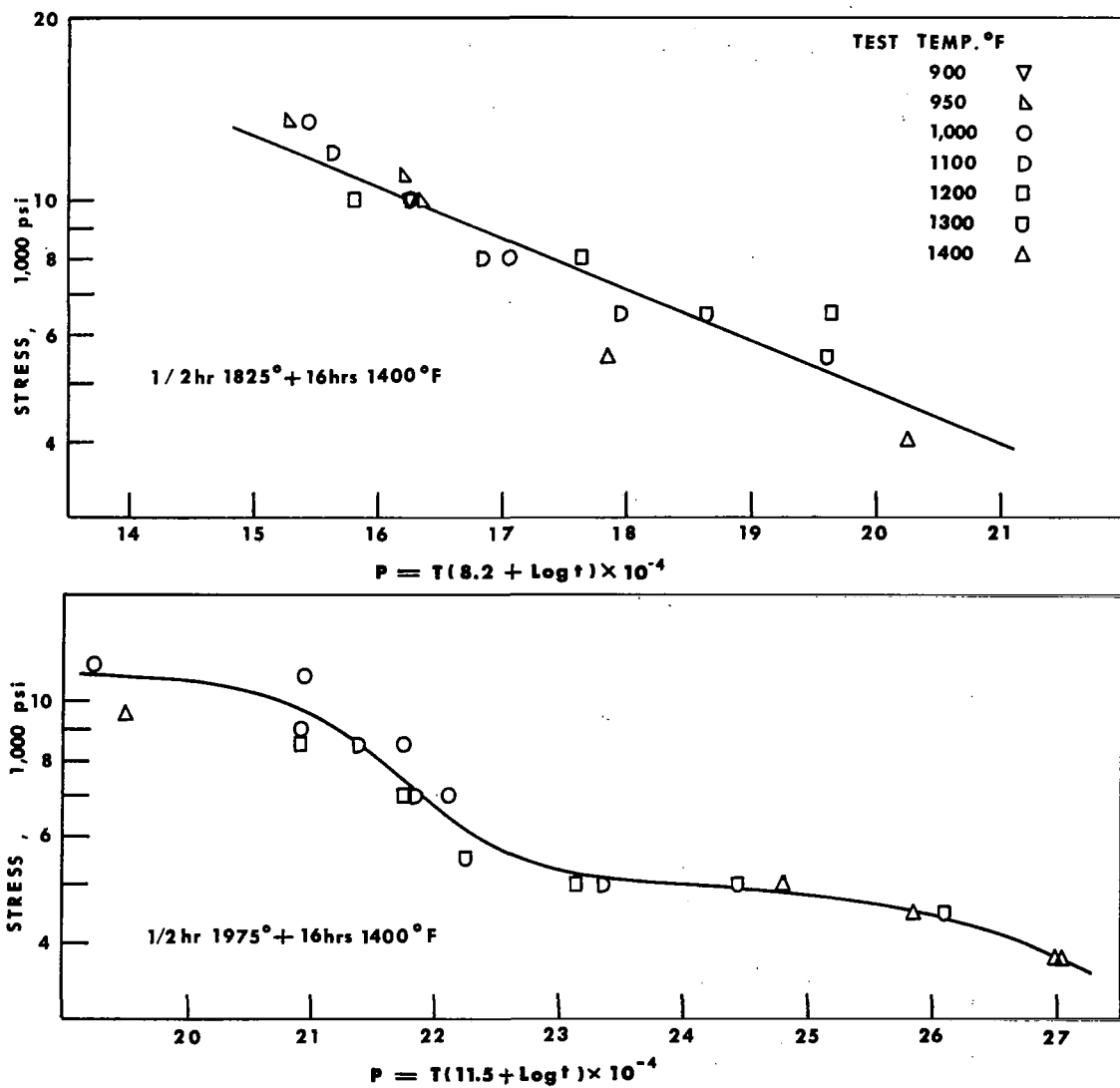


Figure 67. Time-temperature dependence of the rupture strengths at temperatures from 900° to 1400°F of notched specimens of 0.026-inch thick Waspaloy sheet heat treated 1/2 hour at 1825°F and aged, and 1/2 hour at 1975°F and aged.

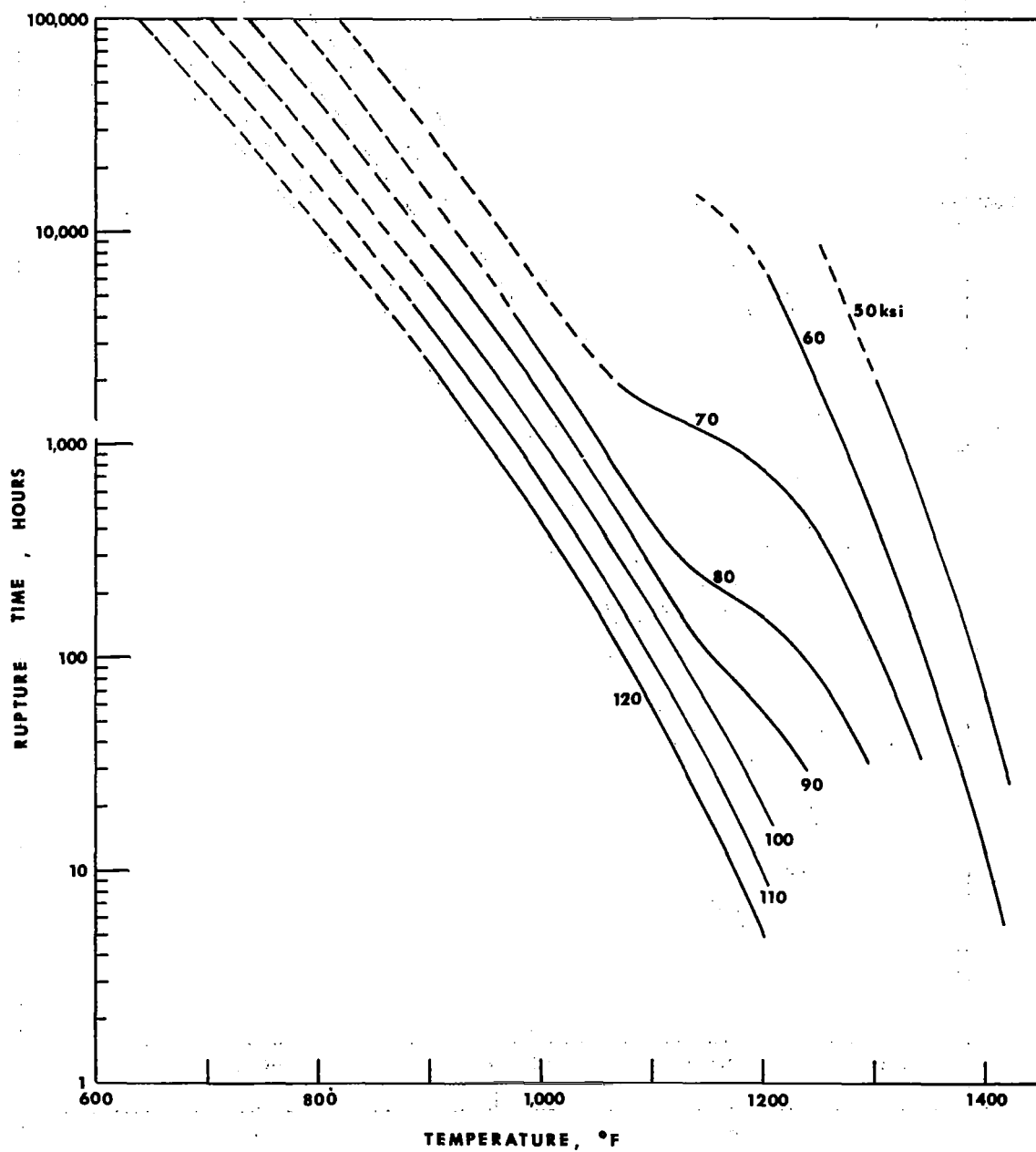


Figure 68. Iso-stress curves of rupture time versus temperature for notched specimens of 0.026-inch thick Waspaloy sheet heat treated 1/2 hour at 1825°F and aged 16 hours at 1400°F.

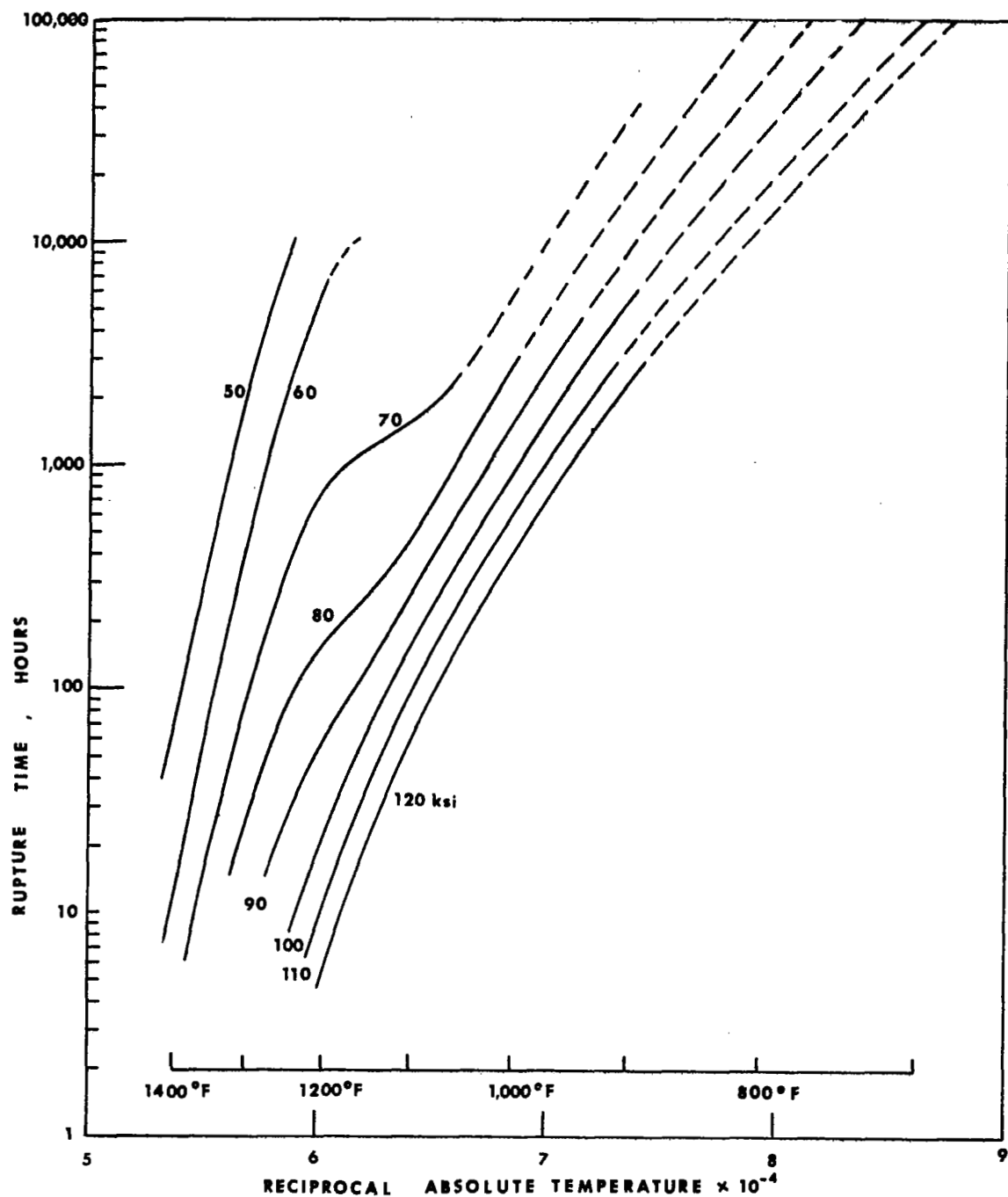


Figure 69. Iso-stress curves of rupture time versus reciprocal absolute temperature for notched specimens of 0.026-inch thick Waspaloy sheet heat treated 1/2 hour at 1825°F and aged 16 hours at 1400°F.

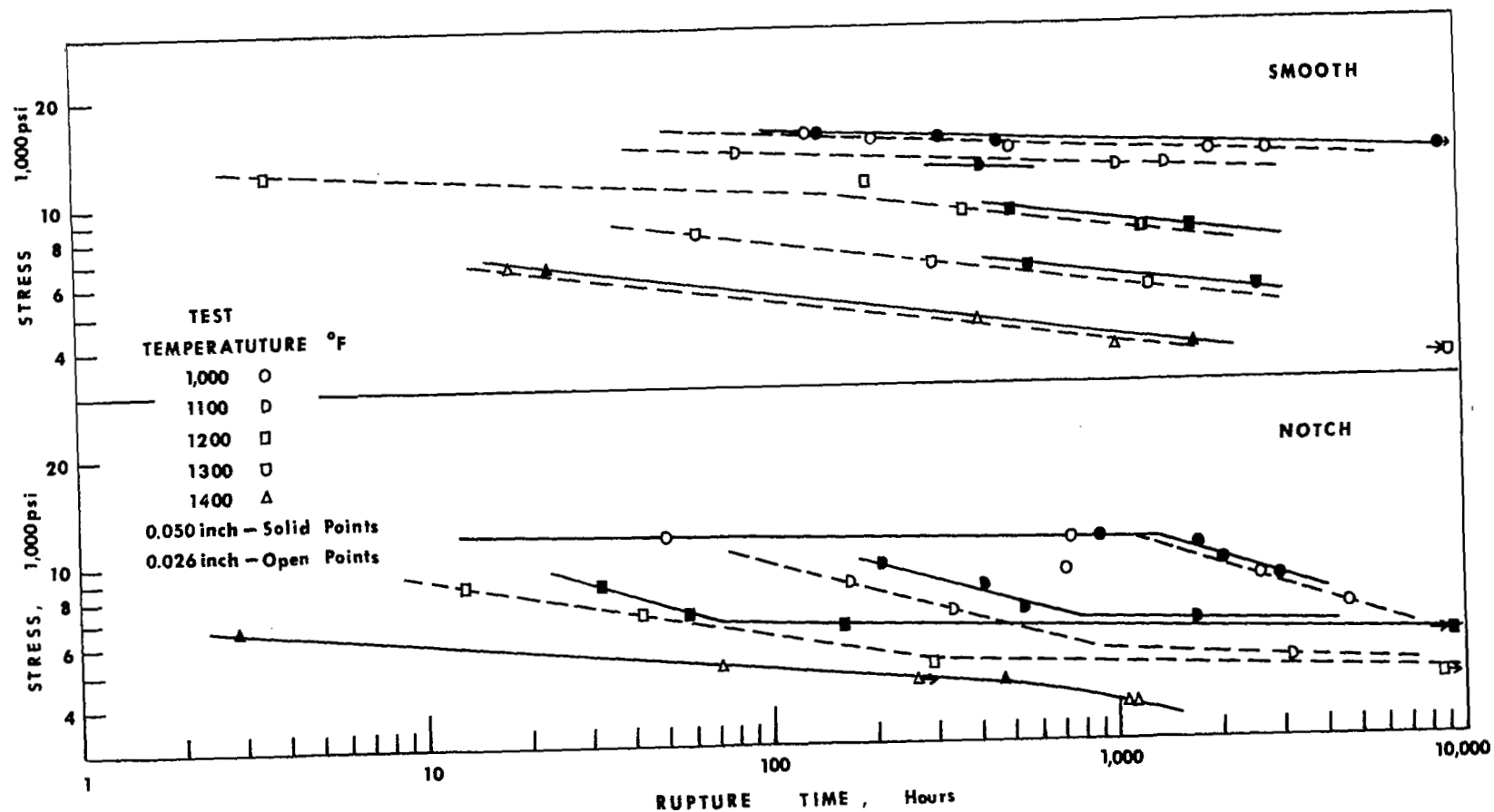


Figure 70. Stress versus rupture time data at temperatures from 1000° to 1400°F for notched and smooth specimens of 0.050 and 0.026-inch thick Waspaloy sheet heat treated 1/2 hour at 1975°F and aged 16 hours at 1400°F.

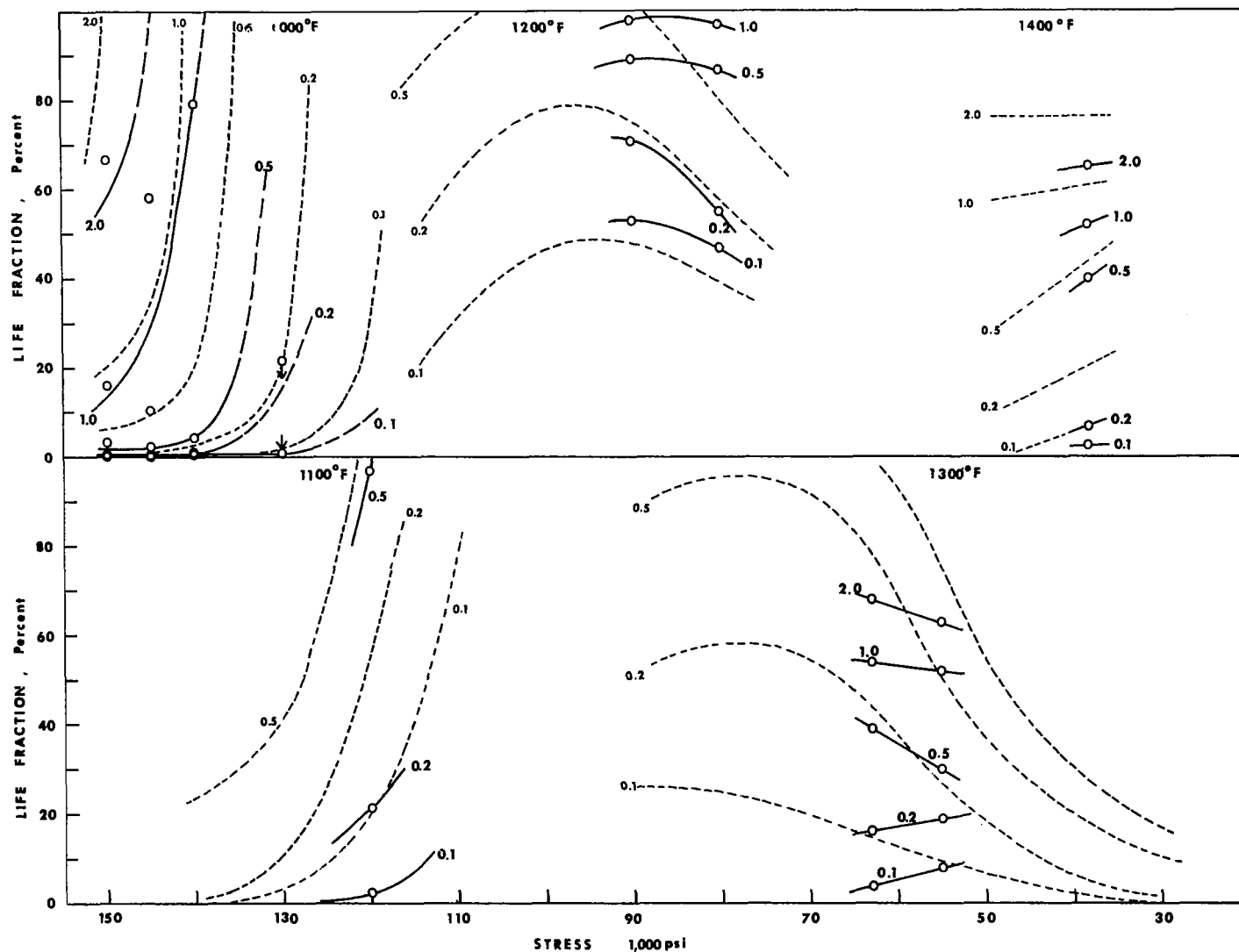


Figure 71. Iso-creep strain curves of life fraction versus stress at temperatures from 1000° to 1400°F for 0.050-inch thick Waspaloy sheet solution treated 1/2 hour at 1975°F and aged 16 hours at 1400°F. The dashed lines are the curves for 0.026-inch material in the identical heat treated condition (see Figure 32).

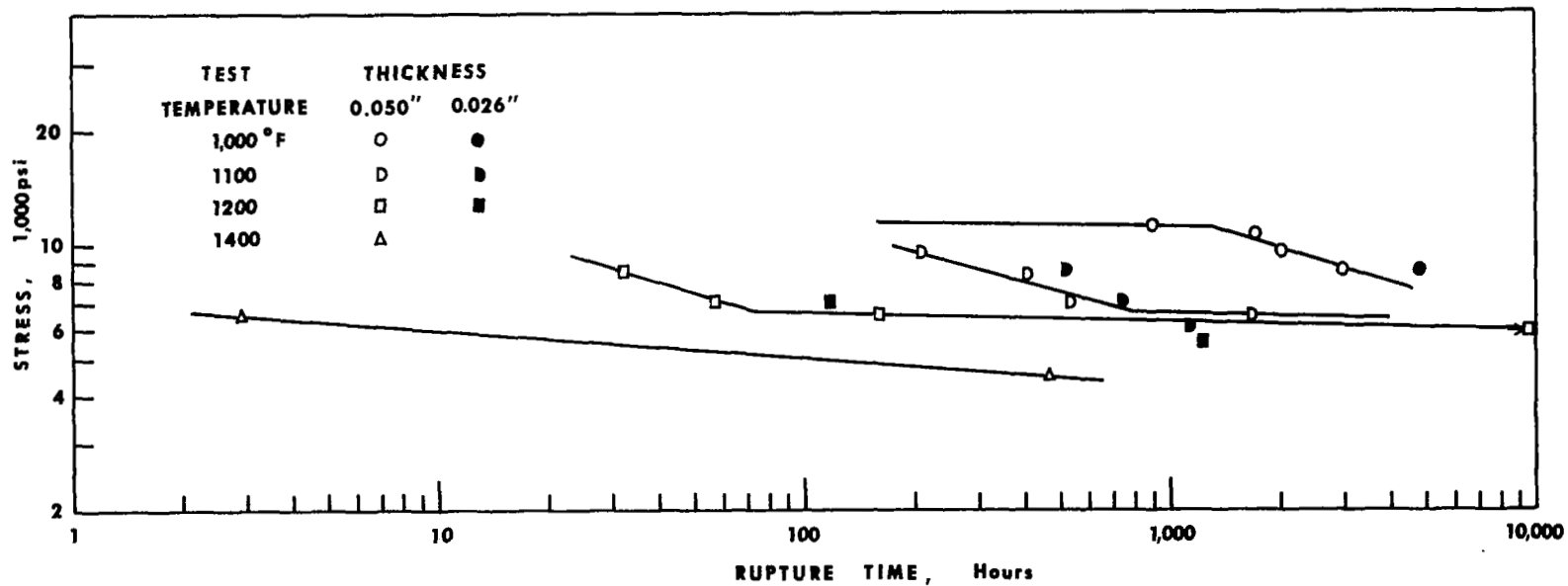


Figure 72. Stress versus rupture time data at temperatures from 1000° to 1400°F for notched specimens of 0.050 and mechanically thinned 0.026-inch thick Waspaloy sheets heat treated 1/2 hour at 1975°F and aged 16 hours at 1400°F.